# ANGLO SAXON CHRONOLOGY PROJECT LABORATORY AND QUALITY ASSURANCE PROCEDURES AT THE QUEEN'S UNIVERSITY, BELFAST RADIOCARBON DATING LABORATORY FOR DATED SAMPLES

# SCIENTIFIC DATING REPORT

Gerry McCormac, Paula Reimer, Alex Bayliss, Michelle Thompson, Nancy Beavan, David Brown, and Stephen Hoper







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#### SUMMARY

The laboratory procedures are described that were used to produce 180 radiocarbon measurements at the Queen's University, Belfast Radiocarbon Dating Laboratory between 1999 and 2006. These samples were dated as part of the English Heritage-funded project, *Anglo-Saxon England c AD 570–720: the chronological basis.* 

Ninety-five of the samples were human bone from graves containing diagnostic artefact assemblages. The remainder of the dated material comprised decadal samples of knownage Irish oak spanning the period AD 390–810. The oak measurements were carried out to provide a bespoke section of calibration curve to ensure maximum accuracy of the calibrated dates on the human remains. Two independent measurements were made on each decadal sample of oak and these were counted quasi-simultaneously with the human bones.

Further radiocarbon and stable isotope measurements undertaken during this period on samples from other projects of relevance to this study are also reported

#### CONTRIBUTORS

Gerry McCormac, Paula Reimer, Alex Bayliss, Michelle Thompson, Nancy Beavan, David Brown, and Stephen Hoper

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#### ARCHIVE LOCATION

Queen's University, Belfast

#### DATE OF PROJECT

1999-2009

#### CONTACT DETAILS

University of Stirling, Stirling, FK9 RLA, Scotland Gerry McCormac: email: PrincipalPA@stir.ac.uk

Queen's University Belfast, School of Geography, Archaeology and Palaeoecology, Belfast, BT7 INN, Northern Ireland Paula Reimer, Michelle Thompson, David Brown, and Stephen Hoper

English Heritage, I Waterhouse Square, 138-142 Holborn, London, ECIN 2ST, England Alex Bayliss Department of Anatomy and Structural Biology, Otago School of Medical Sciences, University of Otago, PO Box 913, Dunedin, New Zealand Nancy Beavan

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## I INTRODUCTION

The methods used for conventional radiocarbon dating by liquid scintillation spectrometry at the Queen's University, Belfast Radiocarbon Dating Laboratory (hereafter Belfast) are described in this report. It outlines the sample pre-treatment methods and describes the measures taken to safeguard the accuracy and the reproducibility of the measurements produced as part of the English Heritage funded research project, *Anglo-Saxon England c AD 570–720: the chronological basis.* Radiocarbon determinations are identified by a unique laboratory code. So, for example, UB is the code for the Queen's University Belfast, and UB-4512 is the 4,512<sup>th</sup> measurement produced by the laboratory. This code is the internationally-agreed identifier by which every radiocarbon determination can be traced.

Bayesian chronological modelling (Bronk Ramsey 1995; Buck *et al* 1996) has been used to combine the high-precision radiocarbon dates of human bone with sequences produced by correspondence analysis of the artefact types recovered from graves (Greenacre 1984; 1992; Høilund Nielsen 1995). The overall aim is to provide accurate estimates of archaeological chronology which span only a few decades (at 95% probability) and thereby verify the calendar dating suggested by typology. The rate of change in atmospheric <sup>14</sup>C in the Anglo-Saxon period makes such resolution feasible, although only within a context of precise and accurate radiocarbon measurement.

## 2 LABORATORY TECHNIQUES

Samples of human bone from skeletons included in the Anglo-Saxon project and decadal wood samples from tree-ring series, which have been dated by dendrochronology to the corresponding time period, were pre-treated to remove contamination and combusted to generate carbon dioxide (CO<sub>2</sub>), which was then converted to benzene (C<sub>6</sub>H<sub>6</sub>). A phosphor is added to the benzene and radioactive disintegrations are counted by measurement of the scintillations resulting from  $\beta$ -particles interacting with the phosphor as the radiocarbon in the samples reverts to nitrogen. The number of radioactive disintegrations is measured for both the unknown age sample and a standard material, and from this a radiocarbon age is determined.

## 2.1 Pre-treatment

Bone samples were pre-treated by a method based on Longin (1971). Each sample is broken into small pieces and placed in a 5I beaker under 2% hydrochloric acid (HCI) for demineralisation. The pH is checked daily and, if the acid is consumed, it is decanted and replaced with fresh acid. The process typically takes 5 days. The liquid is then discarded and the solid residue is washed with deionised water to remove calcium ions thus helping to prevent the formation of humates. The sample is then placed in 0.01M HCI and heated to 80–90°C for 5–18 hours to extract collagen in the lysing process. The liquid containing the collagen is then vacuum filtered through glass microfibre paper (70mm GF/D grade)

(Fig 1) before being poured into an evaporating basin and dried in a moisture extraction oven at 100°C (Fig 2). A repeat lysing step is performed on the solid residue to extract further collagen from the sample. The pH of the dried protein is raised (a modification to the Longin method) by reconstituting the sample in deionised water. This precipitates acid-soluble solid impurities before the sample is again dried in a moisture extraction oven.

Wood samples were chemically pre-treated to holocellulose using the method outlined by Green (1963). The samples from each decade are cut into matchstick size pieces using a hammer and chisel or planed before being granulated in a Wiley intermediate mill (20 mesh). They are placed in acidified sodium chlorite (NaClO2) solution ('bleach') overnight before being drained and heated in 4% HCl for one hour. Samples are then vacuum filtered through glass microfibre paper using deionised water until neutral pH after which they are dried in a moisture extraction oven.



Figure I: Vacuum filtration of the liquid containing the collagen (photo Amanda Grieve)



Figure 2: Collagen after moisture extraction (photo Amanda Grieve)

## 2.2 Combustion

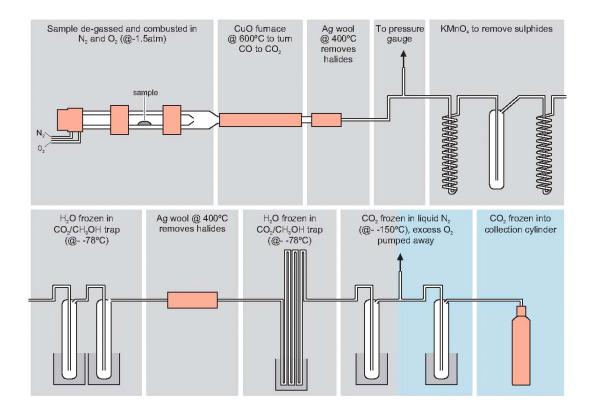
The pre-treated material is combusted in a stream of ultra pure oxygen in a positive pressure system (*c* 1.5atm) that has been purged by ultra pure nitrogen (this prevents the sample from being contaminated by atmospheric CO<sub>2</sub> which contains a small fraction of radiocarbon) (Fig 3). The resultant gas is passed through a furnace containing copper oxide at 600°C to convert any carbon monoxide (CO) present to CO<sub>2</sub>. The gas is purified by the removal of halides at two stages by means of silver wool at 400°C. Other volatile gaseous by-products are oxidised and removed by passing the gas through three traps containing a saturated solution of potassium permanganate (KMnO<sub>4</sub>). Water is removed from the gas using three 'cold' traps of dry ice-methanol mixture. The purified and dried CO<sub>2</sub> is collected in a trap under liquid nitrogen (Fig 4). Glass-metal liners provide a temperature gradient that limits the amount of carrier oxygen frozen, allowing a longer combustion time into the trap and faster evacuation of excess oxygen when the system is opened to vacuum. The frozen CO<sub>2</sub> is expanded and trapped in a metal storage vessel ('bomb'). A schematic diagram summarising the combustion process is provided in Figure 5.



Figure 3: Sample combustion (photo Amanda Grieve)



Figure 4: Freezing the CO2 in a liquid N2 trap (@~ -150°C) (photo Amanda Grieve)



*Figure 5: Schematic diagram summarising the steps in the combustion process (John Vallender)* 

## 2.3 Fractionation Correction

A sub-sample of the CO<sub>2</sub> is taken to measure the ratio of <sup>13</sup>C to <sup>12</sup>C in a mass spectrometer (VG 602e Micromass). In nature, there is a tendency for the lighter carbon isotope (<sup>12</sup>C) to be taken up preferentially, and so plants and animals may have a lower <sup>14</sup>C concentration than the atmosphere. This effect is known as fractionation (Bowman 1990, 20). Fractionation may also occur during laboratory handling due to incomplete reactions. The ratio of <sup>12</sup>C to <sup>13</sup>C ( $\delta^{13}$ C) is measured with respect to an internationally agreed standard (VPDB; Craig 1953; 1957), and is usually expressed in parts per thousand (per mil.; ‰). As <sup>13</sup>C does not decay radioactively, the <sup>13</sup>C/<sup>12</sup>C ratio remains constant, and can be used to estimate the original uptake of <sup>14</sup>C, because the fractionation of <sup>14</sup>C should be double that of <sup>13</sup>C. The  $\delta^{13}$ C value is incorporated into the calculation of the radiocarbon age (Stuiver and Polach 1977). If this correction was not made, the radiocarbon measurement could be anomalously old.

## 2.4 Benzene Synthesis

The CO<sub>2</sub> is converted in an evacuated system to the carbon-rich liquid, benzene (C<sub>6</sub>H<sub>6</sub>) (Fig 6).



## Figure 6: Benzene synthesis (photo Amanda Grieve)

A sequence of reactions is involved. First the  $CO_2$  reacts with molten ultra pure lithium (Li) at 600°C in a vessel (316 steel) according to the equation:

 $2CO_2 + 12Li \rightarrow Li_4C_2 + 4Li_2O$ 

Radon (222Rn) free water is then added to the cooled vessel, producing acetylene ( $C_2H_2$ ) and by-products as follows:

 $Li_4C_2 + 4H_2O \rightarrow C_2H_2 + 4LiOH + H_2$ 

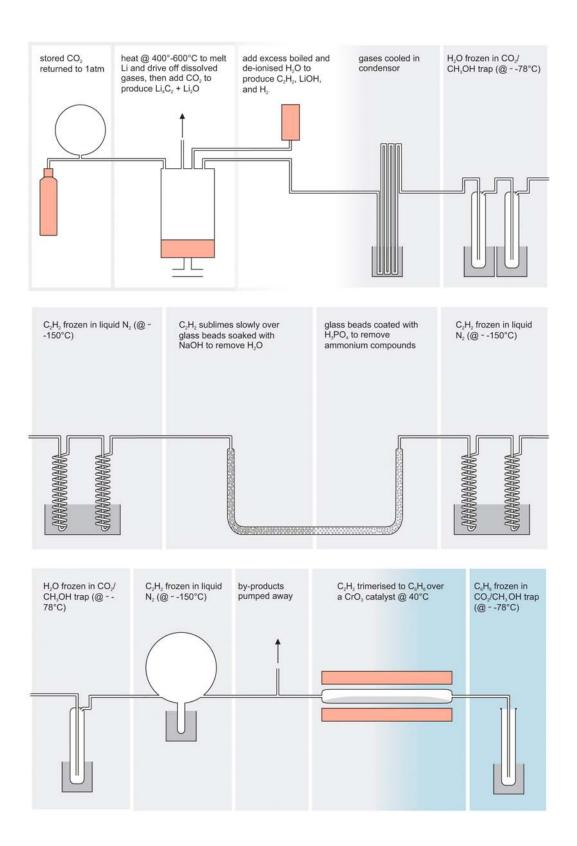
Water vapour is removed from the resultant  $C_2H_2$  in a dry ice-methanol cold trap. The  $C_2H_2$  is then frozen and the hydrogen (H2) is removed under vacuum. The  $C_2H_2$  is then allowed to sublime slowly through beads coated with sodium hydroxide (NaOH), which removes any remaining moisture, and then orthophosphoric acid ( $H_3PO_4$ ), which removes

ammonium compounds. The final step is the trimerisation of the  $C_2H_2$  to  $C_6H_6$  which is accomplished over a chromium oxide (CrO<sub>3</sub>) catalyst at 40°C (Fig 7). The benzene is collected and stored for at least three weeks to allow any <sup>222</sup>Rn to decay. This is a contaminant radio-isotope with a short half-life which derives from the catalyst.

A schematic diagram summarising the conversion of the  $CO_2$  to  $C_6H_6$  is provided in Figure 8.



Figure 7: Loading the chromium oxide catalyst to convert the acetylene to benzene (photo Amanda Grieve)



*Figure 8: Schematic diagram summarising the steps in the benzene synthesis process (John Vallender)* 

## 2.5 Liquid Scintillation Counting

Once the radon has decayed, the sample benzene is thawed and transferred into a lowpotassium glass vial for counting in the liquid scintillation spectrometer. Vials are already pre-selected for each counter according to optical quality, uniformity of glass and weight range, and all have undergone tests using backgrounds and oxalic acid standards. They have been matched with a counting cap that minimizes weight loss during the counting period (McCormac et al 2001). A precise amount of scintillant (butyl-PBD) is required according to the weight of benzene. This phosphoric compound dissolves to create a 'counting cocktail' and produces a light emission (photon) when a  $\beta$ - particle is emitted by a <sup>14</sup>C atom as it decays. The sample is transferred to the counting vial under a fume hood using a pipette/syringe and a five figure balance (Fig 9). It is essential for the calculation of the radiocarbon age that the weights of the benzene and the scintillant are known precisely. For this reason specially designed vial caps are used at every stage to minimize loss of the volatile benzene by evaporation (Pearson 1983). Counting is not started on the prepared vials until the day after the scintillant is added to the benzene, and they are re-weighed before they are loaded into a counter, to ensure that there are no undetected leaks from the vials before counting begins. Weights are monitored weekly throughout the counting period to track benzene loss.

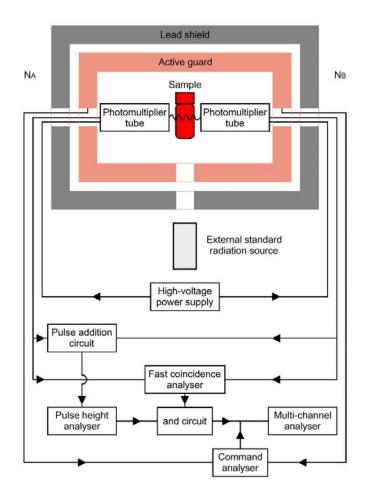


Figure 9: Preparing the benzene sample for counting (photo Amanda Grieve)

Vials are placed in the liquid scintillation spectrometer along with known-activity and background standards (Fig 10). All samples were counted in LKB Wallac 1220 scintillation spectrometers. These optimised, low-level, high precision counters are set up and closely monitored for stability and performance (McCormac 1992; Wilson *et al* 1995; McCormac *et al* 2001). A schematic diagram of a counter is shown in Figure 11. Samples are counted alongside the international reference material, Oxalic Acid II (Mann 1983). Radiocarbon-free ultra pure benzene (from a petro-chemical source) is counted in each machine to monitor background activity. Each spectrometer counts two modern and two background standards. Counts from these standards are used in the calculation of the resulting radiocarbon age.



Figure 10: Loading prepared vials into the liquid scintillation spectrometer (photo Amanda Grieve)



#### Figure 11: A schematic diagram of a liquid scintillation spectrometer (John Vallender)

The vial is positioned between two photomultiplier tubes which detect the light emissions. If both tubes register a pulse simultaneously and the energy of the combined pulse is within the expected range for radiocarbon, a count is recorded in a multi-channel analyser.

Passive and active shielding is employed to distinguish between sample counts (derived from the radiocarbon in the sample as it decays) and background counts (derived from background radiation). The detector assembly is enclosed in 0.2m thick lead casing to absorb background radiation. Active shielding is provided by a mineral oil-based scintillator known as the 'guard'. The photons produced by background radiation events are also detected by two photomultiplier tubes within the guard. If these pulses are coincident with counts in the detector assembly, then the sample counts are discarded as they probably came from background radiation.

Two further checks are available to ensure the validity of the detected pulses. First, the energy spectrum of the pulse is measured to ensure that it has not shifted with respect to the spectra obtained from calibration standards. Second, the sample is irradiated by an external radiation source, radium ( $^{226}$ Ra) measured quasi-continuously. This produces a flood of photons with a range of energies. If the benzene has a low level of impurities then the end point of this energy distribution will fall within a specific range. The set-up of counters labelled, Q1, Q3, and Q4 is as described in McCormac (1992), that of counters Q2, Q6, and Q7 is as described in McCormac *et al* (2001).

## 3 CALCULATION OF THE RADIOCARBON AGE

Once a sample has accumulated more than 250,000 counts, an age calculation can be made based on the following equation:

$$T = -\underline{T}_{_{\!\!\!\!\!/_2}} \ln (A^{_{\!\!\!\!/_2}}\!/A^{_{\!\!\!/_0}}\!) \text{ yr} = -\,8033 \ln (A^{_{\!\!\!/_2}}\!/A^{_{\!\!\!/_0}}\!) \text{ yr}$$
  
In 2

where In is the natural logarithm (to base e),  $A^{14}$  is the measured activity of the sample (in counts per minute per gram), and  $A^{14}_{o}$  is the original <sup>14</sup>C activity as defined by the activity of the modern standard (by convention this equation uses a half-life of 5568 years, rather than the actual half-life of 5730 years). Normal background activity is accounted for in the overall calculation as well as background contributed by the processing of the sample through the benzene line (McCormac *et al* 1993). Corrections for fractionation ( $\delta^{13}$ C) and for spectral shifts are also factored in (McCormac 1992).

A conventional radiocarbon age is accompanied by a standard error. This is based on the accumulated counts for the sample, oxalic acid, and background material (Stuiver and Polach 1977). Other corrections are applied, to account for other sources of error introduced, for example, by laboratory processing (Pearson 1980; Pearson *et al* 1986; McCormac *et al* 1993). A laboratory error multiplier (K) is also applied as a measure of the laboratory reproducibility, incorporating the errors resulting from gas preparation, sample loading, memory effects, and counting statistics (Pearson and Stuiver 1986, 839). 'K' is defined as the actual standard error divided by the quoted standard error and is usually generated through replicate measurement of standard or known-age material. For the analyses reported as part of this project a laboratory error multiplier of 1.25 has been applied. This is slightly more conservative than the 1.23 reported for the 1986 calibration data from Belfast (Pearson *et al* 1986).

## 4 CALIBRATION

The rate of production of radiocarbon in the atmosphere is not constant. Long term variations in the production rate correspond with changes in the strength of the earth's magnetic field and short term variations are linked to sunspot activity. The effect of this is that a radiocarbon 'year' is not equivalent to a calendar year and hence a calibration

process is necessary. The ability for absolute and precise dating of naturally occurring materials which exhibit annual growth patterns (eg tree rings) has made calibration of the radiocarbon time scale possible. Calibration curves have been produced and allow conversion between conventional radiocarbon ages and calendar dates. Variation in the concentration of <sup>14</sup>C in the biosphere over time can be seen in the secular variation and 'wiggles' on calibration curves. A general introduction to radiocarbon calibration can be found in Aitken (1990), further details of the currently internationally agreed calibration curve can be found in Reimer *et al* (2004).

As part of the Anglo-Saxon project, wood samples from the time period under study were processed in the laboratory alongside the samples of human bone. As well as addressing the issue of calibration by refining the existing data, the exercise also provides an additional measure of quality control on the samples of unknown age.

## 5 KNOWN-AGE WOOD SAMPLES

Decadal wood samples from a section of the Irish oak tree-ring chronology were measured specifically for the Anglo-Saxon period under study. Bi-decadal samples from this chronology had been previously measured and included in the calibration series (Pearson *et al* 1986). In this study, decadal samples were replicated to ensure internal consistency by dividing blocks of wood into two for separate measurement. In addition, to study the effect of sample combustion, a comparison was made of samples separated before combustion with those separated after combustion. Initially decadal measurements were completed for the period AD 495–725 (McCormac *et al* 2004) which were included in the INTCAL04 calibration curve (Reimer *et al* 2004), and later further measurements were made extending the curve in both directions to cover the period AD 395–805 (McCormac *et al* 2008).

## 5.1 Dendrochronology

In total forty-two 100g samples of oak (*Quercus* spp.) from a series of decades centred on AD 394.5 to AD 804.5 were processed by the laboratory. The majority of the wood samples came from three sites located in the southern part of Ireland. Samples with decades centred between AD 394.5 and AD 564.5 were from Little Island, Co. Cork (51° 53' N and 8° 55' W), except for decade AD 554.5, a replacement sample, which came from Lemanaghan, Co. Offaly (53° 17' N and 7° 45' W). Samples with decades centred between AD 574.5 and AD 724.5 were from Brabstown, Co. Kilkenny (52° 40' N and 7° 24' W). Both the Little Island and the Brabstown samples have been used before in high precision radiocarbon calibration (McCormac *et al* 1994, tables 1 and 2). The extension of this exercise into the eighth and early ninth centuries AD raised some difficulties. This is a period where there are fewer trees available in the Belfast tree-ring chronology. Fewer trees and requests for samples for various radiocarbon calibration exercises over a number of years have lead to reduced availability of the required weight of wood for conventional radiometric dating. Two samples, from a section of chronology that had not previously been used for radiocarbon calibration from Cappagh South, Co. Clare (52° 44' N and 8° 46' W) were used to cover the period from AD 735 to AD 805. Details of the wood samples dated are given in Table 1.

Decade centred	Weight	Sample number	Site name	Date supplied	Laboratory Numbers
AD 804.5	108g	Q9309	Cappagh South	27/01/2004	UB-6121-22
AD 794.5	100g	Q9309	Cappagn South	27/01/2004	UB-6119-20
AD 794.3 AD 784.5		Q9309 Q9309	· · · -	27/01/2004	UB-6117-8
	8g	*	Cappagh South		
AD 774.5	2g	Q9309	Cappagh South	27/01/2004	UB-6115-16
AD 764.5	108g	Q9308	Cappagh South	27/01/2004	UB-6113-14
AD 754.5	100g	Q9308	Cappagh South	27/01/2004	UB-6111-12
AD 744.5	100g	Q9308	Cappagh South	27/01/2004	UB-6109-10
AD 734.5	100g	Q9308	Cappagh South	27/01/2004	UB-6107-8
AD 724.5	105g	Q3693	Brabstown	18/01/1999	UB-4409-4410
AD 714.5	105g	Q3693	Brabstown	18/01/1999	UB-4407–8; UB- 5232
AD 704.5	108g	Q3693	Brabstown	18/01/1999	UB-4405–6
AD 694.5	101g	Q3693	Brabstown	18/01/1999	UB-4403-4
AD 684.5	109g	Q3693	Brabstown	18/01/1999	UB-4401-2
AD 674.5	103g	Q3693	Brabstown	18/01/1999	UB-4399-4440
AD 664.5	103g	Q3693	Brabstown	18/01/1999	UB-4397–8
AD 654.5	104g	Q3693	Brabstown	18/01/1999	UB-4395–6
AD 644.5	101g	Q3693	Brabstown	18/01/1999	UB-4393-4
AD 634.5	108g	Q3693	Brabstown	18/01/1999	UB-4391-2
AD 624.5	105g	Q3693	Brabstown	18/01/1999	UB-4389–90
AD 614.5	108g	Q3693	Brabstown	18/01/1999	UB-4387–8
AD 604.5	88g	Q3693	Brabstown	18/01/1999	UB-4385–6
AD 604.5	17g	Q3693/Q3685	Brabstown	19/01/1999	
AD 594.5	109g	Q3693/Q3685	Brabstown	19/01/1999	UB-4383-4
AD 584.5	63g	Q3693/Q3685	Brabstown	19/01/1999	UB-4381-2
AD 574.5	102g	Q3693/Q3685	Brabstown	19/01/1999	UB-4379-80
AD 564.5	104g	Q3676/Q3679/Q3674	Little Island	26/01/2001	UB-4616–17
AD 554.5	-	Q9795	Lemanaghan	-	UB-4618–19
AD 544.5	168g	Q3676	Little Island	26/01/2001	UB-4620-1
AD 534.5	126g	Q3676	Little Island	26/01/2001	UB-4622–3
AD 524.5	109g	Q3676/Q3681	Little Island	26/01/2001	UB-4624–5
AD 514.5	107g	Q3676/Q3681	Little Island	26/01/2001	UB-4626–7
AD 504.5	106g	Q3676/Q3681	Little Island	26/01/2001	UB-4628–9
AD 494.5	103g	Q3676/Q3681	Little Island	26/01/2001	UB-4630-1
AD 484.5	72g	Q3676	Little Island	18/06/2003	UB-4933-4
AD 474.5	96g	Q3676	Little Island	18/06/2003	UB-4935–6
AD 464.5	108g	Q3676	Little Island	18/06/2003	UB-4937–8
AD 454.5	108g	Q3676	Little Island	18/06/2003	UB-4939–40
AD 444.5	103g	Q3676	Little Island	18/06/2003	UB-4941–2
AD 434.5	100g	Q3676	Little Island	18/06/2003	UB-4943-4
AD 424.5	100g	Q3676	Little Island	18/06/2003	UB-4945-6
AD 414.5	103g	Q3676	Little Island	18/06/2003	UB-4947–8
AD 404.5	103g	Q3676	Little Island	18/06/2003	UB-4949–50
	ι ν 'δ	2000		1010012003	55 1717-50

Table I: Decadal wood samples provided for the Anglo-Saxon chronology project

### 5.1.1 Dendrochronology methodology

Tree-ring dating was carried out using the methodology described by Baillie (1982). The samples were prepared by polishing the surface with various grades of emery paper. The tree-ring pattern was highlighted with chalk so that the spring and summer pores retain the chalk and provide better visual definition of the ring widths under the microscope.

Measurement of the tree-ring pattern was carried out using a measuring stage. This machine measures the annual growth ring widths in 1/50mm. The tree-ring series obtained from the samples were plotted on graph paper for visual comparisons. The data obtained were also checked statistically with the Belfast tree-ring chronology for Ireland and with regional and site chronologies. Correlation values in Table 2 were obtained using the cross-dating program Cross84 (Munro 1984). All the samples used in this exercise had extremely high or very significant correlation values with regional or site master chronologies from Ireland.

Table 2: Tree-ring cross-matches from (***=extremely significant match; **=ve nsm=non-significant match)	,	
Site chronology name	Correlation Value	

Site chronology name	Correlation Value
Little Island, Co. Cork	
Lemanaghan, Co. Offaly (Brown pers comm)	6.03***
Ballydaly, Co. Offaly (Brown pers comm)	5.70***
Nendrum, Co. Down (McErlean and Brown 2007)	5.32***
Teeshan, Co. Antrim (Baillie 1982, 188–90)	4.69**
Brabstown, Co. Kilkenny (Baillie 1982, 188–90)	3.94nsm
Brabstown	
Ballyroe, Co. Wexford (Brown pers comm.)	8.54***
Ballyrafton, Co. Kilkenny (Baillie 1982, 186–92)	7.44***
Newtown, Co. Tipperary (Brown pers comm)	7.18***
Ballygeardra, Co. Kilkenny (Baillie 1982, 186–92)	6.29***
Ballygormill South, Co. Laois (Brown pers comm)	5.98***
Cappagh South, Co. Clare	
Newtown, Co. Tipperary (Brown pers comm)	6.12***
Ballyroe, Co. Wexford (Brown pers comm)	6.04***
Brabstown, Co. Kilkenny (Baillie 1982,186–92)	5.36***
Ballygeardra, Co. Kilkenny (Baillie 1982, 186–92)	4.89***
Lemanaghan, Co. Offaly	
Ballydaly, Co. Offaly (Brown pers comm)	12.96***
Ballykillen, Co.Offaly (Baillie 1982,186–92)	8.03***
Deer Park, Co. Antrim (Brown pers comm)	6.29***
Little Island, Co. Cork (Brown pers comm)	6.05***

-

### 5.1.2 Little Island tree-ring chronology

A mean tree-ring master chronology was formed using the measurements from eight samples obtained from this mill structure. This chronology is 370 years in length. Extremely significant and consistent correlation values were found with a number of site chronologies from southern Ireland (Table 2). These results indicate that the Little Island chronology dates from AD 261 to AD 630.

### 5.1.3 Brabstown tree-ring chronology

A mean master was formed using the measurements of six structural timbers from the Brabstown horizontal mill. This chronology is 333 years in length. It was compared with a series of site chronologies from Ireland as shown in Table 2. Extremely significant and consistent correlation values were found. The results indicate that the Brabstown chronology dates from AD 549 to AD 881.

### 5.1.4 Cappagh South samples

The timbers from Cappagh South, Co. Clare are from two naturally preserved riverside oaks. These series cross-match with each other with a t-value of 7.23 (Munro 1984). Evidence for the dating of the two-timber mean is provided in Table 2. These results indicate that sample Q9308 dates from AD 630 to AD 821, while sample Q9309 dates from AD 745 to AD 895.

#### 5.1.5 Lemanaghan tree-ring chronology

A mean master was formed using the measurements from 10 structural timbers from the Lemanaghan trackways. This chronology is 364 years in length. It was compared with a series of site chronologies from Ireland as shown in Table 2. Extremely significant and consistent correlation values were found. The results indicate that the Lemanaghan chronology dates from AD 288 to AD 652.

## 5.2 Radiocarbon Ages from Known-age Wood

For decades AD 495–805, samples were pre-treated before being divided into pairs for separate combustion; samples from decades AD 395–485 were pre-treated and combusted before pair separation at the benzene synthesis stage (see Section 5.3 below). Each sample from the replicate pairs from the same decade was counted in a separate batch in the spectrometers to maximise the independence of the radiocarbon determinations. A replicate measurement was made for the decade centred on AD 714.5

in 2006 (UB-5232). This sample was excess cellulose from UB-4407–8, pre-treated in 1999, but combusted and synthesised to benzene separately in 2006.

Full details of all the radiocarbon results from the known-age wood samples are provided in Table 3. The results are represented graphically in Figure 12.

			nple I		San	nple 2	
Decade	Site Details	Radiocarbon	error	δ <sup>13</sup> C	Radiocarbon	error	δ <sup>I3</sup> C
Centred		Age (BP)		(‰)	Age (BP)		(‰)
(AD)			20	240		12	240
394.5	Little Island, Co. Cork	1645	20	-24.9	1659	13	-24.9
404.5	Little Island, Co. Cork	1649	20	-25.4	1611	13	-25.4
414.5	Little Island, Co. Cork	1621	19	-24.7	1635	21	-24.7
424.5	Little Island, Co. Cork	1598	19	-25.3	1560	21	-25.3
434.5	Little Island, Co. Cork	1530	19	-24.4	1529	21	-24.4
444.5	Little Island, Co. Cork	1591	19	-24.8	1539	21	-24.8
454.5	Little Island, Co. Cork	1549	19	-23.9	1554	21	-23.9
464.5	Little Island, Co. Cork	1589	21	-24.9	1589	22	-24.9
474.5	Little Island, Co. Cork	1574	21	-25.8	1537	21	-25.8
484.5	Little Island, Co. Cork	1559	21	-26.2	1504	21	-26.2
494.5	Little Island, Co. Cork	1650	21	-25.I	1595	20	-25.0
504.5	Little Island, Co. Cork	1611	21	-25.2	1606	20	-25.0
514.5	Little Island, Co. Cork	1556	21	-24.5	1595	20	-25.0
524.5	Little Island, Co. Cork	1607	21	-24.8	1613	20	-24.7
534.5	Little Island, Co. Cork	1571	20	-24.5	1598	20	-24.5
544.5	Little Island, Co. Cork	1538	20	-24.0	1572	20	-24.3
554.5	Lemanaghan, Co. Offaly	1511	17	-26.9	1534	19	-26.6
564.5	Little Island, Co. Cork	1524	20	-24.5	1510	20	-24.4
574.5	Brabstown	1475	24	-23.8	1499	24	-23.8
584.5	Brabstown	1486	24	-24.8	1464	24	-24.7
594.5	Brabstown	1504	24	-24.6	1442	24	-24.6
604.5	Brabstown	1439	24	-24.0	1478	24	-24.I
614.5	Brabstown	1423	24	-23.8	1452	24	-23.8
624.5	Brabstown	1425	24	-24.3	1451	24	-24.2
634.5	Brabstown	1415	22	-24.3	1432	22	-24.3
644.5	Brabstown	1424	22	-24.7	1425	22	-24.7
654.5	Brabstown	1373	22	-25.0	1371	22	-25.I
664.5	Brabstown	1343	22	-25.0	32	22	-25.4
674.5	Brabstown	1305	22	-26.0	1285	22	-26.0
684.5	Brabstown	1281	24	-25.6	1281	24	-25.5
694.5	Brabstown	1249	24	-25.7	1271	24	-25.8
704.5	Brabstown	1264	24	-25.7	1263	24	-25.9
714.5	Brabstown	1238	22	-25.2	1266	22	-25.2
724.5	Brabstown	1279	22	-25.5	1249	22	-25.5

Table 3: Radiocarbon ages and  $\delta^{3}C$  values for decadal wood samples dated as part of the Anglo-Saxon chronology project

734.5	Cappagh South, Co. Clare	1216	16	-25.I	2	22	-24.9
744.5	Cappagh South, Co. Clare	1294	16	-25.0	1248	23	-25.8
754.5	Cappagh South, Co. Clare	1262	16	-26.3	1268	29	-25.9
		Sam	nple I		San	nple 2	
Decade Centred (AD)	Site Details	Radiocarbon Age (BP)	error	δ <sup>13</sup> C (‰)	Radiocarbon Age (BP)	error	δ <sup>13</sup> C (‰)
764.5	Cappagh South, Co. Clare	1275	16	-26.9	1233	23	_ 25.9
774.5	Cappagh South, Co. Clare	1154	20	-25.0	1116	22	_ 25.0
784.5	Cappagh South, Co. Clare	4	20	-26.2	1143	22	- 26.3
794.5	Cappagh South, Co. Clare	1145	20	-25.8	1195	22	_ 25.8
804.5	Cappagh South, Co. Clare	1184	20	-25.7	1150	22	- 25.7

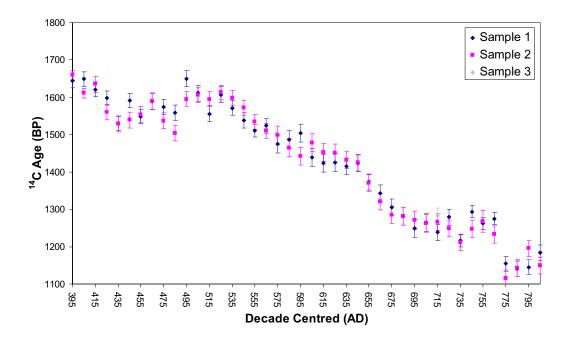
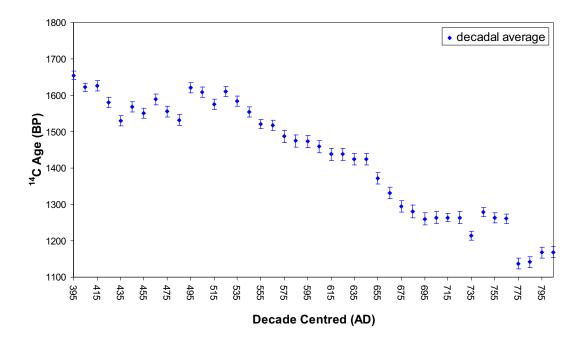


Figure 12: Radiocarbon measurements from decadal samples of Irish oak (McCormac et al 2004; 2008)

	this rep	ort	Stuiver <i>et al</i> (19	98a)	Pearson <i>et al</i> (1986	6)
Decade Centred	Weighted Mean (BP)	error	Radiocarbon Age/ decadal average	error	Radiocarbon Age/ decadal average (BP)	erro
(AD)			(BP)		<b>U</b> ( )	
394.5	1655		1666	13		
404.5	1622		1666	15	1645	17
414.5	1627	14	1639	16		
424.5	1581	14	1618	13	1617	17
434.5	1530	14	1560	16		
444.5	1568	14	1556	16	1571	17
454.5	1551	14	1580	17		
464.5	1589	15	1578	17	1553	17
474.5	1556	15	1560	15		
484.5	1532	15	1561	17	1588	17
494.5	1621	15	1561	13		
504.5	1608	15	1580	15	1552	17
514.5	1576	15	1573	14		
524.5	1610	15	1591	10	1534	18
534.5	1585	14	1559	16		
544.5	1555	14	1497		1534	19
554.5	1521	13	1509	17		
564.5	1517	4	1491	16	1487	18
574.5	1487	17	1497	17		
584.5	1475	17	1475	16	1524	18
594.5	1473	17	1489	17		
604.5	1459	17	1452	15	1463	18
614.5	1438	17	1442	32		
624.5	1438	17	444	16	1402	18
634.5	1424	16	1460	12		
644.5	1425	16	1397	17	1408	12
654.5	372	16	37	16		
664.5	1332	16	1342	17	1329	18
674.5	1295	16	1324	17		
684.5	28	17	33	18	1279	18
694.5	1260	17	1262	16		
704.5	1264	17	1294	15	1282	17
714.5	1264	12	1273	13		
724.5	1264	16	1254	13	1246	12
734.5	1214	13	1248	10		
744.5	1279	13	1263		1278	16
754.5	1263	4	1296	8		
764.5	1261	13	1282	10	1247	16
774.5	37	15	1247			
784.5	1142	15	1168	10	1240	16
794.5	1168	15	1213	8	•	
804.5	1169	15	1197	13		

Table 4: Decadal/bi-decadal averages of radiocarbon measurements from known-age wood samples, AD 395–805



# Figure 13: Decadal means of radiocarbon measurements from decadal samples of Irish oak (McCormac et al 2004; 2008)

Before this project two sets of measurements were already available on known-age wood for the Anglo-Saxon period. Bi-decadal samples of Irish oak (*Quercus* spp.) were measured in Belfast in the early 1980s (Pearson *et al* 1986) and decadal samples of sequoia (*Sequoia dendron*) and Douglas fir (*Pseudotsuga menziesii*) from the western coast of North America were measured by the University of Washington, Seattle (Stuiver and Becker 1986; corrected as described by Stuiver *et al* 1998a).

The decadal means of the data from this study are plotted with respect to Pearson *et al* (1986) in Figure 14<sup>2</sup>. To investigate differences between datasets, weighted means were calculated from the four decadal measurements on each bi-decade previously sampled. These twenty bi-decadal means were compared with the original Belfast measurements. In six of the twenty cases the pairs are statistically significantly different, with three pairs being different at more than 99% confidence. The mean difference between these datasets is  $-10.7\pm4.4$  yr BP (Table 5).

# Table 5: Differences between the bi-decadal measurements on Irish oak (Pearson et al 1986) and the decadal measurements on Irish oak (Table 3)

Number of comparisons = 20 Average difference = -9.7Average standard deviation in difference = 19.9Mean difference =  $-10.7 \pm 4.4$ Standard deviation (=square root of sample variance) = 33.7k = Standard deviation/Average standard deviation in difference = 1.7

Bi-decade centred (AD)	Difference (yr BP)	Standard Deviation
409.5	-20.8	19.1
429.5	-61.8	19.7
449.5	-11.6	19.7
469.5	18.8	20.0
489.5	-10.7	19.9
509.5	40.4	19.9
529.5	62.9	20.6
549.5	2.3	21.2
569.5	17.8	21.0
589.5	-50.0	21.6
609.5	-15.0	21.6
629.5	28.1	21.4
649.5	-9.8	16.3
669.5	-15.5	21.1
689.5	-8.5	21.6
709.5	-18.2	20.6
729.5	-11.5	15.6
749.5	-6.3	18.7
769.5	-40.4	18.8
789.5	-85.3	9.

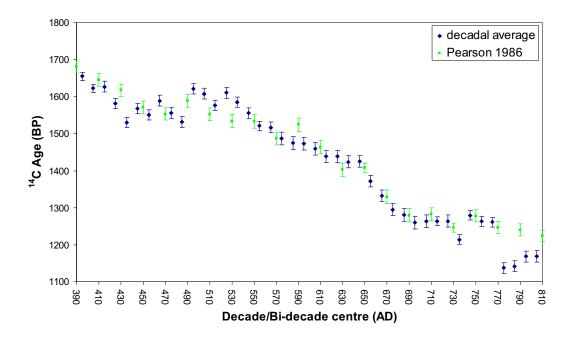


Figure 14: Means of radiocarbon measurements from decadal samples of Irish oak (McCormac et al 2004; 2008) and bi-decadal samples of Irish oak (Pearson et al 1986)

The new data are plotted alongside the measurements on North American conifers by the Seattle group (Stuiver and Becker 1986; corrected as described by Stuiver *et al* 1998a) in Figure 15. Eight decadal pairs are statistically significantly different in this case, with four pairs being different at more than 99% confidence. The mean difference between these datasets is  $-10.3\pm3.1$  yr BP (Table 6).

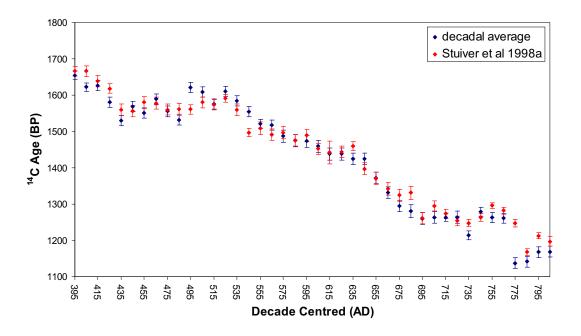


Figure 15: Means of radiocarbon measurements from decadal samples of Irish oak (McCormac et al 2004; 2008) and decadal samples of American conifer (Stuiver and Becker 1986, corrected as reported in Stuiver et al 1998a)

Table 6: Differences between the decadal measurements on American conifers (Stuiver et al 1998a) and the decadal measurements on Irish oak (Table 3)

Number of comparisons = 42 Average difference = -9.1Average standard deviation in difference = 21.0 Mean difference =  $-10.3 \pm 3.1$ Standard deviation (=square root of sample variance) = 32.4 k = Standard deviation/Average standard deviation in difference = 1.6

Decade Centred (AD)	Difference (yr BP)	Standard Deviation
394.5	-11.2	17.0
404.5	-43.7	18.5
4   4.5	-11.7	21.3
424.5	-37.1	19.2
434.5	-30.4	21.3
444.5	11.6	21.3
454.5	-28.7	22.1
464.5	11.0	22.8
474.5	-4.5	21.1
484.5	-29.5	22.5
494.5	60.2	19.5
504.5	28.4	20.9

514.5	3.4	20.2
Decade Centred (AD)	Difference (yr BP)	Standard Deviation
524.5	19.1	17.6
534.5	25.5	21.3
544.5	58.0	17.9
554.5	12.2	21.2
564.5	26.0	21.3
574.5	-10.0	24.0
584.5	0.0	23.3
594.5	-16.0	24.0
604.5	6.5	22.7
614.5	-4.5	36.2
624.5	-6.0	23.3
634.5	-36.5	19.7
644.5	27.5	23.1
654.5	0.1	22.3
664.5	-10.0	23.1
674.5	-29.0	23.1
684.5	-50.0	24.8
694.5	-2.0	23.3
704.5	-30.5	22.7
714.5	-9.3	17.9
724.5	10.0	20.3
734.5	-33.7	16.3
744.5	16.0	7.
754.5	-32.6	6.1
764.5	-20.7	6.5
774.5	-110.2	18.4
784.5	-26.1	17.9
794.5		16.8
804.5	28.4	19.7

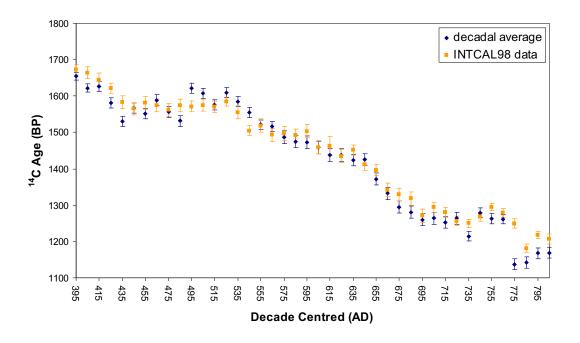


Figure 16: Means of radiocarbon measurements from decadal samples of Irish oak (McCormac et al 2004; 2008 and IntCal98 (Stuiver et al 1998b)

Table 7: Differences between the decadal values for IntCal98 (Stuiver et al 1998b) and the decadal measurements on Irish oak (Table 3)

Number of comparisons = 42 Average difference = -13.7Average standard deviation in difference = 21.7Mean difference =  $-14.6 \pm 3.3$ Standard deviation (=square root of sample variance) = 32.0k = Standard deviation/Average standard deviation in difference = 1.5

Decade Centred (AD)	Difference (yr BP)	Standard Deviation
394.5	-17.3	18.1
404.5	-41.3	19.8
414.5	-17.9	22.5
424.5	-40.0	20.2
434.5	-53.1	22.5
444.5	2.3	22.4
454.5	-30.0	22.6
464.5	14.7	23.3
474.5	-6.3	22.3
484.5	-42.8	23.1
494.5	50.5	21.0
504.5	33.1	21.7
514.5	5.9	21.5
524.5	25.1	18.8

Decade Centred(AD)	Difference (yr BP)	Standard Deviation
534.5	29.5	22.6
544.5	50.4	19.4
554.5	2.2	22.4
564.5	23.8	22.7
574.5	-10.3	25.1
584.5	-17.1	24.2
594.5	-30.4	24.8
604.5	0.7	23.9
614.5	-24.8	30.7
624.5	2.8	24.4
634.5	-28.6	20.8
644.5	13.4	22.1
654.5	-23.6	21.5
664.5	-9.7	24.0
674.5	-34.3	24.0
684.5	-36.7	25.3
694.5	-10.5	24.7
704.5	-30.7	22.2
714.5	-17.4	18.3
724.5	7.9	20.6
734.5	-36.1	16.8
744.5	10.9	17.9
754.5	-32.0	17.4
764.5	-16.7	17.9
774.5	-12.4	19.6
784.5	-39.1	18.6
794.5	-50.5	17.5
804.5	-38.4	20.6

Considering the decades in which the available calibration measurements are statistically significantly different, only in two cases are the new data incompatible with all three comparative datasets: Pearson *et al* (1986), Stuiver *et al* (1998a), and IntCal98 (Stuiver *et al* 1998b). These are for the decades centred on AD 775 and AD 795. The results of Pearson *et al* (1986) and Stuiver *et al* (1998a) are consistent for the former, but statistically significantly different for the latter. The new results show a rapid enrichment of <sup>14</sup>C between AD 765 and AD 775, which lies outside the 95% probability envelope for IntCal04 (Reimer *et al* 2004). Some of the decadal measurements made at the University of Washington (Stuiver *et al* 1998a) confirm atmospheric <sup>14</sup>C enrichment in this interval, although in a few instances these measurements lie closer to the original bi-decadal Belfast measurements. The large number of samples processed for this time period may suggest that there is difficulty in reaching a consensus value, or that there are regional differences in the interval of rapid enrichment. Further work is required to resolve this issue. The variability in the different calibration datasets vindicates the methodology of replicating sections of the calibration curve specifically for this project.

Figure 17 shows the radiocarbon age from Grave 91 at Edix Hill, Cambridgeshire (UB-4512; 1345±18 BP) calibrated using the raw measurements from the three calibration datasets currently available for the seventh century AD. The differences in the probability distributions of the calibrated dates are relatively subtle (less than twenty calendar years). However, because the rate of change of <sup>14</sup>C at this time produces precise calibrated dates, these differences can be proportionally significant. Variations of this magnitude are of considerable importance in the archaeological and historical interpretation of Anglo-Saxon chronological sequences.

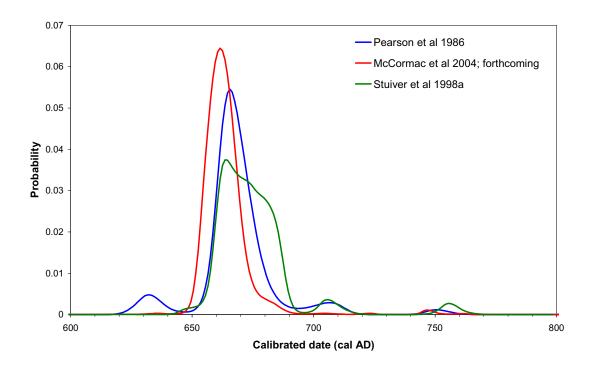


Figure 17: Probability distributions for UB-4512 (1345±18 BP), calibrated by the probability method (Stuiver and Reimer 1993), using data from Pearson et al (1986), McCormac et al (2004), and Stuiver et al (1998b)

## 5.3 The effect of pair separation

As noted above, to study the effect of sample combustion, a comparison was made of samples separated before combustion (decades AD 494.5–804.5, UB-4616–31, UB-4379–4410, and UB-6107–22) with those separated after combustion but before benzene synthesis (decades AD 394.5–484.5, UB-4933–52).

Table 8 shows the difference between replicate measurements on samples which were divided before combustion. The mean difference between these 34 pairs is  $-0.8\pm$  5.2 yr BP. Table 9 shows the difference between replicate measurements on samples which were divided after combustion, but before benzene synthesis. The mean difference between these 10 pairs is 18.0  $\pm$  8.7 yr BP. Although this may appear to show a

difference in results as a consequence of the sample preparation method, these differences are not statistically significant.

It should be noted that, because the samples were split after the production of CO<sub>2</sub>, only one  $\delta^{13}$ C measurement was made for each decadal pair in the range AD 394.5–484.5 (UB-4933–52).

# Table 8: Differences between paired measurements on known-age wood (Table 3)separated before combustion

Number of comparisons = 34 Average difference = -1.1Average standard deviation in difference = 30.7Mean difference =  $-0.8 \pm 5.2$ Standard deviation (=square root of sample variance) = 30.2k = Standard deviation/Average standard deviation in difference = 1.0

Decade Centred (AD)	Difference (sample 1-sample 2) (yr BP)	Standard deviation
494.5	55	29.0
504.5	5	29.0
514.5	-39	29.0
524.5	-6	29.0
534.5	-27	28.3
544.5	-34	28.3
554.5	-23	25.5
564.5	14	28.3
574.5	-24	33.9
584.5	22	33.9
594.5	62	33.9
604.5	-39	33.9
614.5	-29	33.9
624.5	-26	33.9
634.5	-17	31.1
644.5	-	31.1
654.5	2	31.1
664.5	22	31.1
674.5	20	31.1
684.5	0	33.9
694.5	-22	33.9
704.5		33.9
714.5 (i)	-28	31.1
714.5 (ii)	-45	29.7
714.5 (iii)	-17	29.7
724.5	30	31.1
734.5	5	27.2

744.5	46	28.0
754.5	-6	33.1
764.5	42	28.0
774.5	38	29.7
784.5	-2	29.7
Decade Centred (AD)	Difference (sample 1-sample 2) (yr BP)	Standard deviation
794.5	-50	29.7
804.5	34	29.7

# Table 9: Differences between paired measurements on known-age wood (Table 3) separated after combustion, but before benzene synthesis

Number of comparisons = 10 Average difference = 18.8 Average standard deviation in difference = 27.9 Mean difference =  $18.0 \pm 8.7$ Standard deviation (=square root of sample variance) = 27.4 k = Standard deviation/Average standard deviation in difference = 1.0

Decade Centred (AD)	Difference (sample 1-sample 2) (yr BP)	Standard deviation
394.5	-14	23.9
404.5	38	23.9
414.5	-14	28.3
424.5	38	28.3
434.5		28.3
444.5	52	28.3
454.5	-5	28.3
464.5	0	30.4
474.5	37	29.7
484.5	55	29.7

# 6 QUALITY ASSURANCE PROCEDURES

Strenuous efforts have been made to ensure the accuracy of the radiocarbon ages and their associated error estimates. In-house procedures involving daily monitoring of spectrometers, weekly monitoring of benzene sample weights, and benzene purity measurements are routinely performed (McCormac 1992; and see Section 2 above). Analyses are regularly carried out on quality control materials such as cellulose. Computerised quality control data are routinely recorded and monitored and age calculations are manually cross-checked.

As a further check to ensure that the methods and safeguards employed within the laboratory are effective, the Belfast laboratory is constantly involved in inter-laboratory comparisons. The laboratory contributed to the third and fourth International Radiocarbon Intercomparison (TIRI and FIRI), results of which are summarised briefly in Table 10. The application, importance, results and conclusions of these trials are detailed by Scott *et al* (1990; 1998) and Scott (2003). As discussed in Section 2, the internationally accepted modern reference material used at Belfast is Oxalic Acid II provided by the National Bureau of Standards (Mann 1983).

Intercomparison studies are useful as indicators of laboratory accuracy for a given level of precision. For the six TIRI samples reported by the Belfast laboratory, all results are statistically consistent with the consensus values (at 95% confidence; Table 10). The Belfast value for TIRI B, a sample of pine dated by tree-ring analysis to 3239–3200 BC, is also consistent with the measurements on the same sample undertaken as part of FIRI (D and F), and with the mean of the two bi-decadal measurements undertaken by Pearson et al (1986) on wood spanning these dates (Fig 18). Of the ten FIRI samples dated at Belfast, eight results are statistically consistent with the consensus values (at 95% confidence; Table 10). The reported age for FIRI H, a sample of oak dated by dendrochronology to 313–294 BC, is identical to the consensus value, although both are slightly older than the majority of the calibration data spanning this period (Fig 19). FIRI I was a sample of pine, dated by dendrochronology to 3299–3257 BC, and converted to cellulose in bulk at the Belfast laboratory. Following soxhlet extraction in petroleum ether, the sample was converted to holocellulose as described by Hoper et al (1998). The Belfast result on this material is significantly younger than the consensus value (at 95% confidence, but not 99%) confidence), and younger than the calibration data available for samples from this period (Fig 20). The other sample whose result varied significantly from the consensus value in this intercomparison was FIRI C. It is uncertain why this result is younger than the consensus value especially since the TIRI F calcite gave an infinite age of >60590, however the FIRI A Kauri wood was at the younger end of the consensus range.

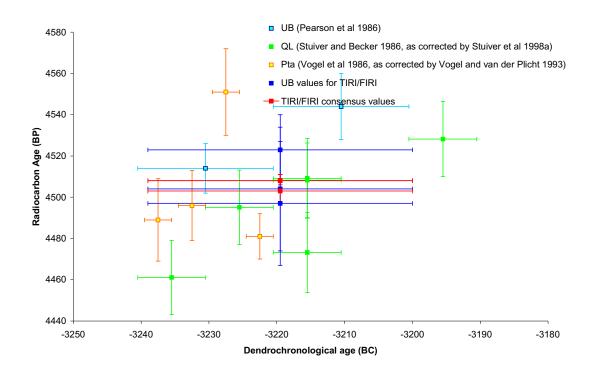


Figure 18: Radiocarbon content of known-age wood samples covering the period of TIRI B and FIRI D and F (3239–3200 BC)

Table 10: Third (TIRI) and Fourth (FIRI) International Radiocarbon Intercomparison results from Queen's University Belfast

TIRI Sample Code and	Consensus Result (all labs) (pMC	QUB Radiometric Result (pMC or
Description	or BP)	BP)
A – barley mash	116.35±0.0084 pMC	116.42 ± 0.20 pMC
B – known-age pine (3239–3200	4503±6 BP	4523 ± 17 BP
BC)		
C – IAEA cellulose	129.7±0.08 pMC	129.56 ± 0.22 pMC
D – Hekla peat	3810±7 BP	3845 ± 22 BP
E – humic acid	11129±0.08 BP	11163 ± 36 BP
F – calcite	>46150	>60590

FIRI Sample Code and	Consensus Result (all labs) (pMC	QUB Radiometric Result (pMC or
Description	or years BP)	Years BP $\pm 1\sigma$ )
A – Kauri wood	0.15 ± 0.44 pMC	0.417 ± 0.023 pMC
B – Kauri wood	0.15 ±0.44 pMC	0.267 ± 0.018 pMC
C – marine turbidite	18173±10.5 BP	17983 ± 50 BP
D – known-age pine (3239–3200	4508±3 BP	4497 ± 30 BP
BC)		
E – humic acid	11778±7 BP	11741 ± 45 BP
F – known-age pine (3239–3200	4508±3 BP	4504 ± 30 BP
BC)		
G – barley mash	110.69±0.04 pMC	110.87 ± 0.35 pMC
H – known-age oak (313–294	2232±5 BP	2232 ± 25 BP
BC)		
I – UB cellulose (3299–3257 BC)	4485±5 BP	4401 ± 40 BP
J - barley mash	110.69±0.04 pMC	111.2 ± 0.35 pMC

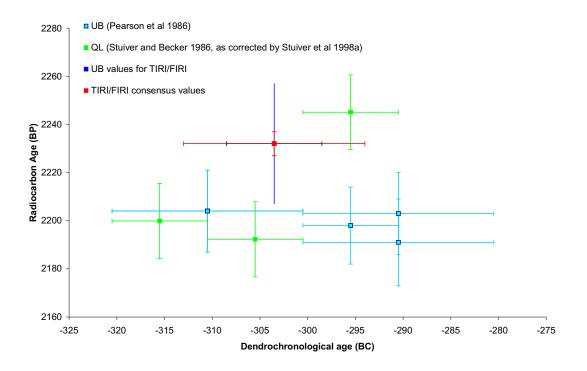


Figure 19: Radiocarbon content of known-age wood samples covering the period of TIRI H (313–294 BC)

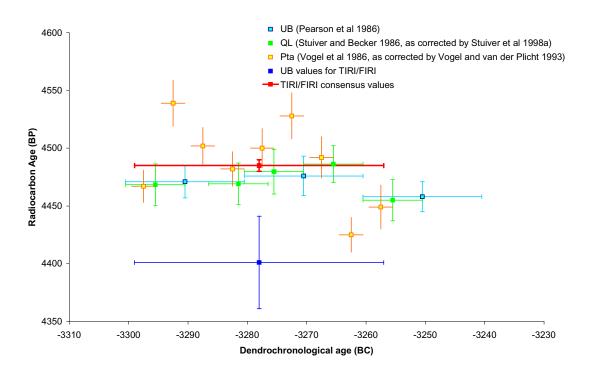


Figure 20: Radiocarbon content of known-age wood samples covering the period of TIRI I (3299–3257 BC)

### 7 BONE SAMPLES

The bone samples were processed in the radiocarbon laboratory as described in Section 2.

One sample, the primary burial from Gally Hills, appeared to have been treated with some form of consolidant between its excavation in 1972 and the collection of the sample in 2001. A radiocarbon determination on the right femur of this individual (UB-4727) was measured in December 2001, and produced a date older than that expected from the accompanying assemblage of grave goods. No conservation records or other information about the treatment of this skeleton was available in the site archive at Bourne Hall Museum (Jeremy Harte pers comm 2002). A second measurement was undertaken on the left femur of this individual in October 2003 (UB-4920). A sub-sample of this bone was tested at the Polymer Processing Research Centre at the Queen's University, Belfast (Millar 2003a). The sample was analysed by Fourier Transform Infra Red Spectroscopy (Hummel 1966) and the presence of Polyvinyl Acetate (PVA) was confirmed (Millar 2003a). This substance is used to consolidate archaeological material (UNESCO 1968; Ashley-Smith 1983), but is petroleum-derived and so of infinite radiocarbon age. If it is not completely removed from a sample, it dilutes the  $^{14}C$ concentration in that sample and so makes its radiocarbon age anomalously old. As PVA is soluble in water, much of the original contamination would have been removed by the pre-treatment protocol for bone described in Section 2 and used for UB-4727. However, UB-4920 was also pre-treated using soxhlet extraction as outlined by Bruhn et al (2001). The sequence of solvents used in this case was: tetrahydrofuran (Boiling Point (B.P) 66°C), chloroform (B.P. 62°C), petroleum ether (B.P. 60°C), acetone (B.P. 56°C), methanol (B.P. 65°C) and water (B.P. 100 °C). Each solvent was applied for two cycles (a cycle being the time taken for the solvent to evaporate, condense, fill sample chamber, and siphon off into boiling flask prior to re-evaporation) at a temperature of 50–70°C for the solvents and 100 °C for the final water step. A further test for contamination was undertaken on the bone after soxhlet extraction, again using Fourier Transform Infra Red Spectroscopy (Millar 2003b). This confirmed the removal of a significant amount of the contaminant, although traces may have remained (Figs 21 and 22). Nonetheless, the sample was then processed for radiocarbon dating. UB-4920 is statistically significantly younger than the original measurement, UB-4727 (T'=8.0; T'(5%)=3.8; v=1; Ward and Wilson 1978). It is probable that UB-4920 is a more accurate estimate of the actual radiocarbon age of the primary burial at Gally Hills than UB-4727, although in the light of the contamination problems described here, the radiocarbon date of this individual must be interpreted with some caution.

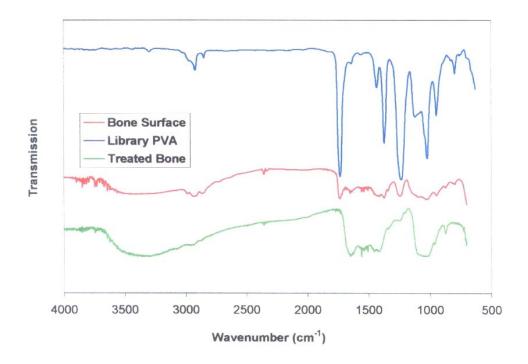
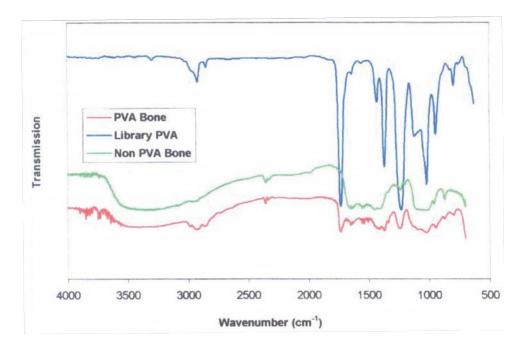


Figure 21: Fourier Transform Infrared Spectroscopy (FTIR) scan of consolidated bone from Gally Hills, and the same bone after soxhlet extraction (Millar 2003b)



# Figure 22: Fourier Transform Infrared Spectroscopy (FTIR) scan of consolidated bone from Gally Hills, and an unconsolidated bone (Millar 2003c)

As a further measure of quality control and to decrease the quoted error on a number of measurements, fourteen benzene samples (UB-4501–11, UB-4551–2, and UB-4554)

were vacuum distilled and recounted in a second liquid scintillation spectrometer, using the standards specific to the new machine. In all cases, the radiocarbon results were statistically consistent and have been combined to produce the radiocarbon ages quoted in Table 11.

Table II: Radiocarbo	on ages and $\delta'^{3}C$ values $^{3}$ for bone s	amples dated as part of the
Anglo-Saxon chronol	logy project (in black), and relevant	t data from other projects (in
blue)		

Laboratory Number	Site & context	Radiocarbon Age (BP)	δ <sup>13</sup> C (‰)
Female Graves	•		
UB-4077	Buttermarket, grave 4275	1476±24	-21.0±0.2
UB-4501	Lechlade: grave 14	32 ±2	-20.0±0.2
UB-4502	Lechlade: grave 138	39 ± 8	-20.4±0.2
UB-4503	Lechlade: grave 148	1319±18	-19.3±0.2
UB-4504	Lechlade: grave 179	1374±20	-20.3±0.2
UB-4506	Lechlade: grave 172/2	1352±19	-20.2±0.2
UB-4507	Lechlade: grave 187	1398±19	-20.2±0.2
UB-4511	Edix Hill: grave 90	1507±19	-20.3±0.2
UB-4512	Edix Hill: grave 91	345± 8	-20.5±0.2
UB-4549	Marina Drive: grave C7	1328±19	-20.6±0.2
UB-4550	Marina Drive: grave El	1379±19	-20.0±0.2
UB-4551	Marina Drive: grave E2	1325±19	-20.3±0.2
UB-4552	Marina Drive: grave E3	1370±19	-20.5±0.2
UB-4553	Marina Drive: grave D10	326±2	-20.8±0.2
UB-4554	Marina Drive: grave F2	1337±19	-20.8±0.2
UB-4705	West Heslerton: grave 123	1502±21	-20.6±0.5
UB-4706	West Heslerton: grave 118	1395±20	-20.2±0.5
UB-4707	Edix Hill: grave 79	1528±21	-20.6±0.5
UB-4708	Edix Hill: grave 83	1488±21	-20.4±0.5
UB-4709	Edix Hill: grave 14	1495±21	-20.7±0.5
UB-4728	Mill Hill: grave 64	1496±22	-19.8±0.5
UB-4729	Mill Hill: grave 68	1503±22	-19.7±0.5
UB-4732	Mill Hill: grave 94	1561±20	-20.0±0.5
UB-4733	Mill Hill: grave 95	1606±20	-20.1±0.5
UB-4734	Mill Hill: grave 105C	1587±19	-20.5±0.5
UB-4735	Berinsfield: grave 22	1567±19	-20.1±0.5
UB-4739	Berinsfield: grave 134/1	1561±21	-20.5±0.5
UB-4835	Appledown Compton, grave 134	1503±16	-20.5±0.5
UB-4836	Westgarth Gardens, grave 27	1560±20	-20.1±0.5
UB-4883	Melbourne: SK1038, SG95	1416±20	-20.4±0.5
UB-4885	Melbourne, InLI 189 SG78 (stratigraphically later than MelSG077)	1479±20	-20.3±0.5
UB-4887	Melbourne: SK 1229 SG82	42 ±20	-20.6±0.5
UB-4888	Melbourne: SK1271 SG89	1536±19	-20.6±0.5
Laboratory Number	Site & context	Radiocarbon Age (BP)	δ <sup>13</sup> C (‰)
, Female Graves			<u> </u>

UB-4889	Melbourne: SK1293, SG69	1459±19	-20.4±0.5
UB-4890	Melbourne: SKI 307 SG75	1548±20	-20.6±0.5
UB-4910	Bloodmoor Hill: grave 22	365± 5	-20.5±0.5
Laboratory Number	Site & context	Radiocarbon Age (BP)	δ <sup>13</sup> C (‰)
UB-4959	Dover Buckland: grave 391A	1420±20	-20.5±0.5
UB-4963	St Peter's Tip: grave 208	432±2	-20.1±0.5
UB-4964	Coddenham: grave 30	4 7± 6	-20.7±0.5
UB-4965	Appledown Compton: grave 117	475±2	-20.9±0.5
UB-4975	Aston Clinton: grave 12	1517±19	-21.0±0.5
UB-4984	Lechlade: grave 18	1507±20	-20.7±0.5
UB-5208	Appledown Compton: grave 107	48 ±20	-20.6±0.5
UB-6032	St Peter's Tip: grave 73	422± 7	-21.2±0.5
UB-6033	West Heslerton: grave 113	497± 7	-21.1±0.5
UB-6034	Castledyke South: grave 120	1502±17	-21.2±0.5
UB-6035	Castledyke South: grave 96	1517±15	-21.6±0.5
UB-6036	Castledyke South: grave 13	42 ± 7	-21.2±0.5
UB-6037	Castledyke South: grave 134	544± 4	-20.9±0.5
UB-6038	Castledyke South: grave 183	449± 4	-21.3±0.5
UB-6040	Castledyke South: grave 53	1535±15	-21.4±0.5
UB-6041	Castledyke South: grave 182	1515±15	-20.8±0.5
	(stratigraphically earlier than UB-6038)		
UB-6042	Castledyke South: grave 88	323± 3	-20.6±0.5
UB-6472	Dover Buckland: grave 222	1550±19	$-20.1 \pm 0.5$
UB-6473	Dover Buckland: grave 250	1572±22	-20.1±0.5
UB-6476	Dover Buckland: grave 339	1592±17	-20.0±0.5
HAR-8243	West Heslerton: grave 177	1610±40	-20.3
Male Graves			
BM-640	Sutton Hoo: grave I (beeswax)	1427±45	-25.0
			(assumed)
BM-688	Sutton Hoo: grave I (wood)	1256±45	-26.6
UB-4074	Buttermarket: grave 2297	1419±23	-20.6±0.2
UB-4422	Sutton Hoo: grave 17 (man)	1534±35	$-20.0\pm0.2$
UB-4423	Sutton Hoo: grave 17 (horse)	420±28	$-22.9\pm0.2$
UB-4505	Lechlade: grave 172/1	383± 9	-20.2±0.2
UB-4508	Edix Hill: grave 12	488± 9	$-20.2\pm0.2$
UB-4509	Edix Hill: grave 33	52 ± 8	$-20.1\pm0.2$
UB-4510	Edix Hill: grave 48	479± 9	-20.2±0.2
UB-4922	replicate of UB-4510	1508±19	$-20.9\pm0.5$
UB-4682	Westgarth Gardens: grave 66	49 ± 8	-20.2±0.2
UB-4727	Gally Hills: primary burial	487± 6	-20.6±0.5
UB-4920	replicate of UB-4727	1419±18	$-20.5\pm0.5$
UB-4730	Mill Hill: grave 79	1542±18	-19.4±0.5
UB-4731	Mill Hill: grave 93	1508±18	-20.1±0.5
UB-4736	Berinsfield: grave 28	1526±21	-20.2±0.5
UB-4882	Melbourne: SKI 187 SG80	1378±20	-20.4±0.5
	(stratigraphically later than MelSG079)		
UB-4884	Melbourne: SKI 188 SG79	1404±19	-20.6±0.5
	(stratigraphically later than MelSG078)		

UB-4886	Melbourne: SK1204 SG77	1458±20	-20.3±0.5
UB-6345	replicate of UB-4886	1516±23	-19.8±0.5
UB-4921	Mill Hill: grave 81	560± 6	-20.6±0.5
UB-4923	Edix Hill: grave 7	1572±20	-20.6±0.5
UB-4924	St Peter's Tip: grave 113	26 ± 6	-20.0±0.5
UB-6534	replicate of UB-4924	3  ± 8	-19.7±0.5
Laboratory Number	Site & context	Radiocarbon Age (BP)	δ <sup>13</sup> C (‰)
UB-4925	St Peter's Tip: grave 68	466± 6	-19.7±0.5
UB-4926	St Peter's Tip: grave 212	1537±18	-20.3±0.5
UB-4927	St Peter's Tip: grave 263	47 ± 8	-20.0±0.5
UB-4928	St Peter's Tip: grave 250	458± 8	-20.4±0.5
UB-4929	St Peter's Tip: grave 194	485± 8	-20.3±0.5
UB-4930	St Peter's Tip: grave 42	4 4± 9	-19.7±0.5
UB-6346	replicate of UB-4930	435± 6	-19.3±0.5
UB-4931	St Peter's Tip: grave 318	1498±21	-20.3±0.5
UB-4958	Dover Buckland: grave 375	493± 8	-20.0±0.5
UB-4961	St Peter's Tip: grave 8	447± 7	-19.8±0.5
UB-4962	St Peter's Tip: grave 196	445± 6	-20.2±0.5
UB-4976	Ford, Laverstock	464± 6	-20.6±0.5
UB-4981	Lechlade: grave 183	469± 8	-20.6±0.5
UB-4982	Lechlade: grave 155	36 ± 7	-21.0±0.5
UB-4683	Lechlade: grave 40	362± 7	-20.1±0.2
UB-4985	Westgarth Gardens: grave 11	1528±18	-21.1±0.5
UB-6039	Castledyke South: grave 94	4 2± 4	-20.8±0.5
UB-6347	Lakenheath: warrior 4222	1640±20	-20.1±0.5
UB-6348	Lakenheath: horse 4206	6  ±20	-22.9±0.5
UB-6474	Dover Buckland: grave 264	528± 7	-20.0±0.5
UB-6475	Dover Buckland: grave 323	49 ± 8	$-20.1\pm0.5$
UB-6477	Dover Buckland: grave 414	1570±20	$-20.1\pm0.5$
UB-6478	St Peter's Tip: grave 360	4 4± 6	-20.4±0.5
UB-6479	Mill Hill: grave 40	1555±22	-19.8±0.5
Comparanda (men)			•
UB-4039	Buttermarket: grave 3871	1441±20	$-20.9\pm0.2$
UB-4642	West Heslerton: grave 72	487± 9	-19.9±0.2
UB-4880	Lundenwic (BOB91)	3 8± 8	-20.4±0.5
HAR-8242	West Heslerton: grave 176	1510±40	-20.6
Comparanda (women)			
UB-4042	Buttermarket: grave 1674	1407±20	-21.3±0.2
UB-4044	Buttermarket: grave 4152	1413±21	-20.3±0.2
OxA-6792	replicate of UB-4044	1200±40	-19.5
OxA-6793	replicate of UB-4044	1355±45	-19.7
OxA-7859	replicate of UB-4044	1365±40	-19.5
OxA-7860	replicate of UB-4044	1415±40	-19.6
UB-4960	Dover Buckland: grave 391B	6  ± 8	-20.3±0.5

### 8 **REPLICATION**

The unknown age bone samples dated for this project were analysed between 1999 and 2006. As discussed in Section 5, 42 pairs of radiocarbon measurements on decadal samples of known-age wood were also undertaken as part of this project. All pairs are statistically consistent (Ward and Wilson 1978). These wood samples were dated in 1999, 2001, and 2003–4, and so demonstrate the reproducibility of the results from the laboratory over an extended period of time.

The sample size required for high-precision radiocarbon dating inevitably limited the number of replicate measurements that could be undertaken on samples of human bone from this project. In five cases, however, replicate samples were dated from bones of the same individual (Table 11). Of these, the two measurements on the primary burial from Gally Hills (UB-4727 and UB-4920) are not statistically consistent, almost certainly because of the difficulty of removing the chemical contamination discussed above. The two results from Edix Hill, grave 48 (UB-4510 and UB-4922) are statistically consistent (T'=1.2; T'(5%)=3.8; v = 1; Ward and Wilson 1978). Replicate measurements on SG77 from Melbourne Water Lane (UB-4886 and UB-6345) are also consistent (T'=3.6; T'(5%)=3.8; v = I); as are the two determinations on St Peter's Tip, grave 42 (UB-4930 and UB-6346; T'=0.7; T'(5%)=3.8; v = I). Finally, the two measurements from St Peter's Tip, grave 113 (UB-4924 and UB-6534) are not statistically consistent (T'=4.3; T'(5%)=3.8; v = I). Although these results are statistically different at 95% confidence, they are consistent at 99% confidence. A further replicate measurement, on the skeleton from Appledown Compton, grave 117 (UB-4965), failed in laboratory processing. The consistency of these replicate pairs on bone samples confirms the reproducibility of the quoted ages over the duration of the project.

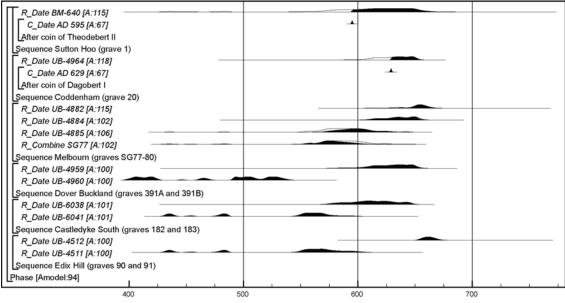
To test for potential dietary offsets in radiocarbon dates obtained from human bone, a pair of measurements was made on a horse and a man buried together in grave 104 at Lakenheath, Suffolk. These results are statistically consistent (UB-6347–8; T'=1.1; T'(5%)=3.8; =1), suggesting that there was no significant marine or freshwater offset in the diet of the dated human being.

Full details of all the radiocarbon results from the bone samples dated as part of this project are provided in Table 11 (in black)<sup>4</sup>. Details of further stable isotope and amino-acid analyses on these samples are provided in Beavan *et al* (2011).

## 9 ARCHAEOLOGICAL AND NUMISMATIC SEQUENCES

Ten samples come from graves whose relative dating is known by archaeological stratigraphy. This information can be used as an independent check on the accuracy of the radiocarbon dating of these skeletons.

Grave 90 from Edix Hill (UB-4511) was overlain by grave 91 (UB-4512) (Malim and Hines 1998, 80–1), and indeed the two measurements are in good agreement with this relative order of burial (Fig 23).



Posterior density estimate (cal AD)

Figure 23: Probability distributions of dates from stratigraphically related burials, and from burials containing coins that can be assigned to documented Merovingian kings. Each distribution represents the relative probability that an event occurs at a particular time. For each of the dates two distributions have been plotted: one in outline, which is the result of simple radiocarbon calibration (McCormac et al 2004; 2008), and a solid one, based on the chronological model used. The large square brackets down the left-hand side along with the OxCal keywords define the overall models exactly

Grave 182 from Castledyke South (UB-6041) was cut by grave 183 (UB-6038) (Drinkall and Foreman 1998, 89 and fig 44), and again the two measurements are in good agreement with this relative order of burial (Fig 23).

Grave 391B from the cemetery at Dover Buckland (UB-4960) was cut by grave 391A (BU-4959) (Parfitt pers comm), and again the two measurements are in good agreement with this relative order of burial (Fig 23).

A sequence of four burials has been dated from Melbourne, Water Lane (Duncan *et al* 2003, 92–4). Two statistically consistent determinations are available from a skeleton buried in the earliest grave, SG77 (UB-4886 and UB-6345). This was cut by grave SG78 which contained the skeleton dated by UB-4885. Grave SG78 was itself cut by grave SG79, which also contained an intact inhumation (UB-4884). Grave SG79 was in turn cut by another grave, SG80 (UB-4882). A chronological model which incorporates this stratigraphic information with the calibrated radiocarbon dates from these burials has good agreement (Fig 23).

Finally, two of the Anglo-Saxon skeletons which have been radiocarbon dated and are included in this project were buried with coins that can be assigned to Merovingian kings who appear in historical sources. Grave 20 at Coddenham, Suffolk (UB-4964) includes a coin of Dagobert I, King of the Merovingian Franks (reigned AD 629–39), and so must date after AD 629, the earliest possible minting date for the coin. Similarly, the coin of Theodebert II, King of Austrasia (reigned AD 595–612) found in the purse which accompanied the ship burial in mound I at Sutton Hoo (Bruce-Mitford 1975, 609), provides a *terminus post quem* of AD 595 for the radiocarbon date from that burial undertaken by the British Museum laboratory in the late 1960s (BM-640). In both cases, the radiocarbon dates from these burials are fully compatible with the historically-attested coin dates (Fig 23).

## 10 $\delta^{13}$ C MEASUREMENTS

For all samples dated at Belfast  $\delta^{13}$ C values are measured for the purposes of age calculation (Stuiver and Polach 1977). As described in Section 2.3, the carbon dioxide produced for dating by the positive pressure flow-through combustion system was subsampled for stable isotope measurement. The fractionation reported in these results might derive from the combustion process in addition to the natural isotopic composition of the dated material. This measurement reflects the true fractionation in the dated sample and so is appropriate for age calculation.

As part of measures to assess the accuracy of the radiocarbon dates for the Anglo-Saxon burials, the natural isotopic composition of the bone samples was of interest, however. In combination, the  $\delta^{13}$ C and  $\delta^{15}$ N values of such bones may be used as an indicator of the proportion of marine protein in an individual's diet in areas such as the British Isles where there were no C4 plants (Mays 1998, fig 9.2). As the marine reservoir is significantly older than the terrestrial biosphere (Hughen *et al* 2004), a diet rich in marine protein may result in a bone with depleted levels of radiocarbon and an older apparent age. Protocols for calibrating such radiocarbon ages accurately, by estimating the proportion of marine and terrestrial components in the diet, are currently inexact (Arneborg *et al* 1999; Bayliss *et al* 2004). In the Anglo-Saxon period, however, a significant proportion of marine protein would not be expected in the diets of most individuals (Privat *et al* 2002), as this predates intensive marine fishing in England (Barrett *et al* 2004a–b). There could have been some marine input into the diets of some dated individuals, however, and so stable

isotope measurements appropriate for inferring past diets were also undertaken as part of this study to check for any ages which might be misleading.

For this reason, sub-samples of the dated bones were sent to the National Isotope Centre, GNS Science, New Zealand. Here they were chemically pre-treated to bone gelatin at the Rafter Radiocarbon Laboratory as described by Beavan-Athfield *et al* (2001). The gelatin was analysed at Isotrace New Zealand for nitrogen and carbon ( $\delta^{15}$ N,  $\delta^{13}$ C, % N and C) using elemental analyser isotope ratio mass spectrometry (EA-IRMS). Full technical details of these analyses are provided in Beavan *et al* (2011).

To investigate the magnitude of fractionation introduced by the positive pressure flowthrough combustion system used for preparing dating samples at Belfast,  $\delta^{13}$ C values were measured by both Belfast and Isotrace New Zealand on sub-samples of the bone protein extracted at Belfast for dating. These replicate pairs of results are listed in Table 12. Figure 24 shows the offset between the results on each sample against the date when the sample was converted to benzene following combustion and stable isotope analysis at Belfast. Before October 2001 the results are in good agreement, with a mean difference of  $0.07\pm0.54\%$ . This negligible difference suggests that minimal additional fractionation is induced in the flow-through combustion system. Bone samples, although more difficult to combust, do not appear to fractionate more during combustion than wood samples, since the mean difference for 30 pairs of wood samples is  $0.57\pm0.22\%$  (Table 13), consistent with that observed for bone samples processed before October 2001. After this date, however, the results on bone protein from Belfast become significantly depleted compared to those made in New Zealand (with a mean difference of  $-1.87\pm1.46\%$ ; Fig 24).

Laboratory	Be	fast	Rafter		Date
Number	<b>δ</b> <sup>13</sup> C (‰)	error (‰)	δ <sup>13</sup> C (‰)	error (‰)	converted to
					$C_6H_6$
UB-4306	-18.6	0.2	-18.7	0.1	01/07/1998
UB-4307	-18.6	0.2	-18.8	0.1	02/07/1998
UB-4308	-18.3	0.2	-18.4	0.1	03/07/1998
UB-4309	-18.3	0.2	-18.6	0.1	06/07/1998
UB-4310	-20.3	0.2	-19.8	0.1	08/07/1998
UB-4549	-20.6	0.2	-21.1	0.1	28/03/2000
UB-4550	-20.0	0.2	-21.1	0.1	29/03/2000
UB-4551	-20.3	0.2	-20.8	0.1	27/03/2000
UB-4552	-20.5	0.2	-22.1	0.1	31/03/2000
UB-4553	-20.8	0.2	-21.1	0.1	04/04/2000
UB-4554	-20.8	0.2	-21.5	0.1	05/04/2000
UB-4641	-20.0	0.2	-20.2	0.1	01/05/2001
UB-4642	-19.9	0.2	-19.8	0.1	02/05/2001

Table 12:  $\delta^{I3}C$  values on the bone protein extracted for radiocarbon dating at the Queen's University, Belfast, measured by (a) the Belfast Radiocarbon Laboratory and (b) Rafter Radiocarbon (samples included in this project are in black)

UB-4643	-20.3	0.2	-20.4	0.1	03/05/2001
Laboratory		Belfast		Rafter	Date
Number	<b>δ</b> <sup>13</sup> C (‰)	error (‰)	δ <sup>13</sup> C (‰)	error (‰)	converted to C <sub>6</sub> H <sub>6</sub>
UB-4644	-21.3	0.2	-19.9	0.1	09/05/2001
UB-4645	-19.9	0.2	-20.4	0.1	/05/200
UB-4646	-20.2	0.2	-20.I	0.1	4/05/200
UB-4647	-   8.8	0.2	-19.1	0.1	6/05/200
UB-4648	-19.9	0.2	-19.8	0.1	17/05/2001
UB-4649	-20.1	0.2	-20.0	0.1	8/05/200
UB-4650	-19.9	0.2	-20.1	0.1	23/05/2001
UB-465 I	-19.4	0.2	-19.2	0,1	24/05/2001
UB-4652	-19.4	0.2	-20.3	0.1	25/05/2001
UB-4653	-20.0	0.2	-20.0	0.1	29/05/2001
UB-4654	-19.7	0.2	-19.9	0.1	01/06/2001
UB-4655	-19.8	0.2	-19.4	0.1	07/06/2001
UB-4656	-20.0	0.2	-20.0	0.1	08/06/2001
UB-4657	-19.5	0.2	-19.0	0.1	22/06/2001
UB-4658	-19.5	0.2	-19.3	0.1	26/06/2001
UB-4659	-19.4	0.2	-19.0	0.1	27/06/2001
UB-4660	-19.5	0.2	-19.5	0.1	28/06/2001
UB-4661	-20.4	0.2	-19.8	0,1	29/06/2001
UB-4662	-20.0	0.2	-19.7	0.1	03/07/2001
UB-4663	-19.2	0.2	-18.8	0.1	06/07/2001
UB-4705	-20.9	0.2	-20.3	0.1	24/10/2001
UB-4706	-21.1	0.2	-20.4	0.1	25/10/2001
UB-4707	-19.7	0.2	-20.2	0.1	26/10/2001
UB-4708	-21.3	0.2	-20.5	0.1	29/10/2001
UB-4709	-21.3	0.2	-20.4	0.1	01/11/2001
UB-4719	-20.8	0.2	-19.7	0.1	05/11/2001
UB-4720	-20.5	0.2	-19.4	0.1	06/11/2001
UB-4727	-21.3	0.2	-20.0	0.1	27/11/2001
UB-4728	-22.1	0.2	-19.9	0.1	28/11/2001
UB-4729	-21.7	0.2	-19.7	0.1	03/12/2001
UB-4730	-20.5	0.2	-19.6	0.1	04/12/2001
UB-4731	-23.1	0.2	-19.4	0.1	05/12/2001
UB-4732	-20.6	0.2	-19.7	0.1	25/01/2002
UB-4733	-21.5	0.2	-19.7	0.1	28/01/2002
UB-4734	-21.2	0.2	-19.9	0.1	29/01/2002
UB-4735	-21.9	0.2	-20.1	0.1	30/01/2002
UB-4736	-22.6	0.2	-20.2	0.1	31/01/2002
UB-4739	-21.9	0.2	-20.5	0.1	01/02/2002
UB-4796	-21.0	0.2	-21.0	0,1	30/04/2002
UB-4797	-20.6	0.2	-20.6	0.1	02/05/2002
UB-4798	-20.7	0.2	-19.6	0.1	10/05/2002
UB-4799	-19.3	0.2	-18.4	0.1	13/05/2002
UB-4800	-21.2	0.2	-20.2	0.1	14/05/2002

UB-4801	-21.8	0.2	-20.3	0,1	15/05/2002
UB-4802	-22.8	0.2	-19.3	0,1	17/05/2002
UB-4803	-22.6	0.2	-19.5	0,1	21/05/2002
UB-4835	-21.3	0.2	-20.4	0.1	24/09/2002
UB-4836	-22.0	0.2	-19.5	0.1	17/09/2002
Laboratory		Belfast		Rafter	Date
Number	<b>δ</b> <sup>13</sup> C (‰)	error (‰)	δ'³C (‰)	error (‰)	converted to C <sub>6</sub> H <sub>6</sub>
UB-4882	-24.6	0.2	-21.3	0.1	20/02/2003
UB-4883	-26.3	0.2	-21.3	0.1	21/02/2003
UB-4884	-21.2	0.2	-21.3	0.1	03/03/2003
UB-4885	-20.9	0.2	-20.9	0.1	05/03/2003
UB-4886	-22.4	0.2	-21.0	0.1	06/03/2003
UB-4887	-22.7	0.2	-21.5	0.1	18/03/2003
UB-4888	-23.9	0.2	-21.4	0.1	14/03/2003
UB-4889	-23.6	0.2	-21.2	0.1	13/03/2003
UB-4890	-23.6	0.2	-21.8	0.1	11/03/2003
UB-4920	-20.1	0.2	-20.2	0.1	25/08/2003
UB-4921	-20.4	0.2	-20.1	0.1	04/07/2003
UB-4922	-23.2	0.2	-20.9	0.1	07/07/2003
UB-4923	-24.4	0.2	-20.6	0.1	23/07/2003
UB-4924	-21.6	0.2	-19.4	0.1	25/07/2003
UB-4925	-20.1	0.2	-19.5	0.1	28/07/2003
UB-4927	-19.4	0.2	-20.2	0.1	19/08/2003
UB-4928	-19.7	0.2	-20.2	0.1	20/08/2003
UB-4929	-19.7	0.2	-19.7	0.1	21/08/2003
UB-4930	-19.7	0.2	-19.2	0.1	27/08/2003
UB-4931	-21.9	0.2	-19.9	0.1	28/08/2003
UB-4961	-20.6	0.2	-18.9	0.1	15/10/2003
UB-4962	-19.8	0.2	-19.0	0.1	16/10/2003
UB-4963	-21.7	0.2	-19.1	0.1	20/10/2003
UB-4964	-20.9	0.2	-19.6	0.1	21/10/2003
UB-4965	-28.8	0.2	-20.7	0.1	22/10/2003
UB-4975	-22.9	0.2	-20.3	0.1	06/11/2003
UB-4976	-21.2	0.2	-19.7	0.1	07/11/2003
UB-4981	-22.2	0.2	-20.1	0.1	26/11/2003
UB-4982	-22.7	0.2	-20.6	0.1	27/11/2003
UB-4984	-22.8	0.2	-19.6	0.1	01/12/2003
UB-4985	-21.1	0.2	-20.2	0.1	08/12/2003
UB-5208	-22.1	0.2	-21.0	0.1	29/09/2005
UB-6032	-21.6	0.2	-19.7	0.1	03/02/2004
UB-6033	-21.8	0.2	-20.0	0.1	04/02/2004
UB-6034	-24.3	0.2	-19.9	0.1	10/03/2004
UB-6035	-24.5	0.2	-20.3	0.1	11/03/2004
UB-6036	-20.6	0.2	-20.3	0.1	25/03/2003
UB-6037	-25.1	0.2	-19.7	0.1	12/03/2004
UB-6038	-23.1	0.2	-19.9	0.1	26/03/2004

UB-6040	-22.2	0.2	-19.9	0.1	20/04/2004
UB-6041	-23.3	0.2	-20.2	0.1	21/04/2004
UB-6042	-21.4	0.2	-19.4	0.1	22/04/2004
UB-6142	-22.8	0.2	-17.2	0.1	26/04/2004
UB-6143	-18.3	0.2	-16.8	0.1	27/04/2004
UB-6144	-19.7	0.2	-17.6	0,1	28/04/2004
UB-6179	-22.1	0.2	-20. I	0.1	01/06/2004
UB-6180	-22.4	0.2	-20. I	0.1	02/06/2004
UB-6181	-24.7	0.2	-20. I	0.1	08/06/2004
Laboratory		Belfast		Rafter	Date
Number	<b>δ</b> <sup>13</sup> C (‰)	error (‰)	δ <sup>13</sup> C (‰)	error (‰)	converted to
					C <sub>6</sub> H <sub>6</sub>
UB-6182	-21.1	0.2	-19.4	0.1	09/06/2004
UB-6183	-21.5	0.2	-19.8	0.1	10/06/2004
UB-6184	-23.7	0.2	-20.0	0.1	14/06/2004
UB-6185	-23.7	0.2	-21.9	0.1	23/04/2004
UB-6345	-22.3	0.2	-19.5	0.1	10/11/2004
UB-6346	-20.7	0.2	-19.0	0.1	09/11/2004
UB-6347	-22.8	0.2	-19.7	0.1	22/11/2004
UB-6472	-22.3	0.2	-19.7	0.1	25/05/2005
UB-6473	-21.4	0.2	-19.5	0.1	26/05/2005
UB-6474	-20.9	0.2	-19.8	0.1	08/06/2005
UB-6475	-20.5	0.2	-19.9	0.1	27/05/2005
UB-6476	-21.3	0.2	-19.6	0.1	30/05/2005
UB-6477	-21.0	0.2	-19.5	0.1	09/06/2005
UB-6479	-20.7	0.2	-19.5	0.1	04/07/2005
UB-6534	-20.0	0.2	-19.1	0.1	29/06/2006

#### pre-October 2001

Number of comparisons = 34 Average difference = 0.05 Average standard deviation in difference = 1.71 Mean difference =  $0.07 \pm 0.04$ Standard deviation (=square root of sample variance) = 0.54 k = Standard deviation/Average standard deviation in difference = 2.40

### post-October 2001

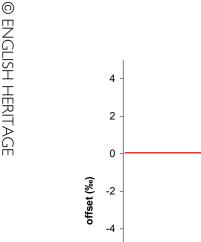
Number of comparisons = 91 Average difference = -1.87 Average standard deviation in difference = 1.46 Mean difference =  $-1.87 \pm 0.02$ Standard deviation (=square root of sample variance) = 1.46 k = Standard deviation/Average standard deviation in difference = 6.54

Laboratory Number	$\delta^{13}$ C (‰) flow-through	$\delta^{13}$ C (‰) closed-tube
	combustion	combustion
UB-4385	-23.980	-23.554
UB-4386	-24.124	-23.554
UB-4389	-24.306	-23.881
UB-4390	-24.215	-23.881
UB-4391	-24.343	-23.774
UB-4392	-24.338	-23.774
UB-4393	-24.715	-24.189
UB-4394	-24.709	-24.189
UB-4395	-24.984	-24.203
UB-4396	-25.091	-24.203
UB-4397	-25.012	-24.415
UB-4398	-25.418	-24.415
UB-4399	-25.982	-25.459
UB-4400	-25.957	-25.459
UB-4401	-25.556	-25.395
UB-4402	-25.503	-25.395
UB-4407	-25.229	-24.477
UB-4408	-25.208	-24.477
UB-4409/4410	-25.457	-24.807
UB-5070	-24.537	-24.055
UB-5072	-24.940	-24.385
UB-5076	-25.141	-24.718
UB-5080	-25.349	-24.449
UB-5082	-24.816	-24.47
UB-5084	-25.337	-24.813
UB-5136	-26.667	-25.685
UB-5150	-27.127	-26.216
UB-5160	-25.774	-25.235
UB-5170	-24.056	-23.493
UB-5172	-23.793	-23.417
UB-5174	-23.792	-23.234

Table 13: Replicate  $\delta^{I3}C$  values for wood cellulose, combusted using flow-through combustion and closed tube combustion

Mean difference =  $0.57 \pm 0.01$ 

Standard deviation (=square root of sample variance) = 0.2



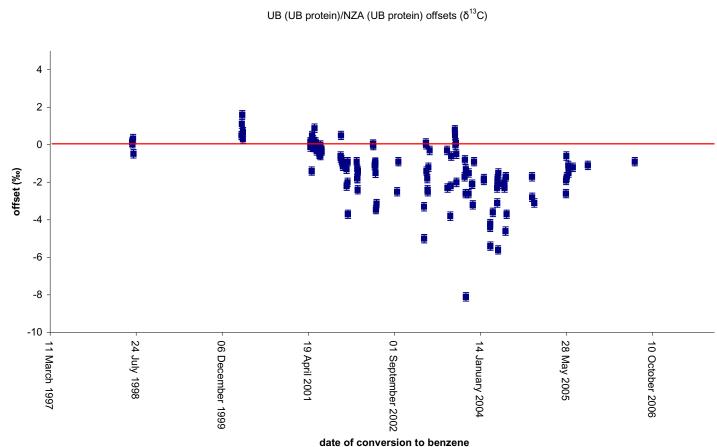


Figure 24: Replicate  $\delta^{I3}C$  values measured by Queen's University Belfast and Isotrace New Zealand on samples of bone protein extracted at Belfast

The difference cannot be attributed to the bone preparation, since  $\delta^{13}$ C values for 40 samples are available from Isotrace New Zealand, on both the protein extracted in Belfast and the gelatin processed at Rafter (Table 14). All these samples were processed for dating after October 2001. The mean difference between these sets of results is 0.49±0.27‰, suggesting that the differences observed in Figure 24 do not derive from methods of sample preparation.

Table 14:  $\delta^{3}C$  values measured by Isotrace New Zealand from bone protein extracted in Belfast and bone gelatin prepared at the Rafter Radiocarbon Laboratory from the same samples

	Isotrace New Ze	aland from Belfast	Isotrace New Zeala	and from Rafter gelatin
	pro	protein		
Laboratory	δ <sup>13</sup> C (‰)	error (‰)	δ <sup>13</sup> C (‰)	total error (‰)
Number				
UB-4961	-18.9	0.1	-19.5	0.32
UB-4962	-19.0	0.1	-19.9	0.32
UB-4963	-19.1	0.1	-19.8	0.32
UB-4964	-19.6	0.1	-20.4	0.32
UB-4965	-20.7	0.1	-20.6	0.32
UB-4975	-20.3	0.1	-20.8	0.32
UB-4976	-19.7	0.1	-20.4	0.32
UB-4981	-20.1	0.1	-20.3	0.32
UB-4982	-20.6	0.1	-20.7	0.32
UB-4983	-19.9	0.1	-20.7	0.32
UB-4984	-19.6	0.1	-20.4	0.32
UB-4985	-20.2	0.1	-20.8	0.32
UB-5208	-21.0	0.1	-20.3	0.36
UB-6032	-19.7	0.1	-20.9	0.32
UB-6033	-20.0	0.1	-20.8	0.32
UB-6034	-19.9	0.1	-20.9	0.32
UB-6035	-20.3	0.1	-21.3	0.32
UB-6036	-20.3	0.1	-20.9	0.32
UB-6037	-19.7	0.1	-20.6	0.32
UB-6038	-19.9	0.1	-21.0	0.32
UB-6040	-19.9	0.1	-21.1	0.32
UB-6041	-20.2	0.1	-20.5	0.32
UB-6042	-19.4	0.1	-20.3	0.32
UB-6179	-20.1	0.1	-20.4	0.32
UB-6180	-20.1	0.1	-20.4	0.32
UB-6181	-20.1	0.1	-20.0	0.32
UB-6182	-19.4	0.1	-19.9	0.32
UB-6183	-19.8	0.1	-20.4	0.32
UB-6184	-20.0	0.1	-20.0	0.32
UB-6345	-19.5	0.1	-19.5	0.36
UB-6346	-19.0	0.1	-19.0	0.36
UB-6347	-19.7	0.1	-19.8	0.36

UB-6472	107	0.1	10.0	0.36
OD-0472	-19.7	0.1	-19.8	0.50
UB-6473	-19.5	0.1	-19.8	0.36
UB-6474	-19.8	0.1	-19.7	0.36
	Isotrace New Zea	aland from Belfast	Isotrace New Zealan	nd from Rafter gelatin
	pro	tein		
Laboratory	δ <sup>13</sup> C (‰)	error (‰)	δ <sup>13</sup> C (‰)	total error (‰)
Number				
UB-6475	-19.9	0.1	-19.8	0.36
UB-6476	-19.6	0.1	-19.7	0.36
UB-6477	-19.5	0.1	-19.8	0.36
UB-6479	-19.5	0.1	-19.5	0.36
UB-6534	-19.1	0.1	-19.4	0.36

Average difference = 0.44

Average standard deviation in difference = 1.70

Mean difference =  $0.49 \pm 0.27$ 

Standard deviation (=square root of sample variance) = 0.44

k = Standard deviation/Average standard deviation in difference = 0.26

Comparison of the  $\delta^{13}$ C values for wood samples measured during the period of the Anglo-Saxon project with those measured earlier at Belfast suggests that the mass spectrometer produced accurate results across the entire measurement period. McCormac *et al* (1994) report  $\delta^{13}$ C values on 269 samples of Irish bog oak, which have a mean value of -25.9±0.9‰. Forty-eight samples of Irish oak were dated between 1999 and October 2001, which have a mean value of -24.8±0.6‰ (Table 15), and 36 samples were dated after October 2001, which have a mean value of -25.3±0.7‰ (Table 16). These mean values are not statistically significantly different (T'=1.0; T'(5%)=6.0; v=2), suggesting that the  $\delta^{13}$ C values measured on oak samples in Belfast do not vary more than would be expected from natural variability between trees. Internal laboratory standards (IAEA C3 cellulose and NBS Oxalic Acid II) also did not show significant deviations from the expected values.

Laboratory code	Date measured	δ <sup>13</sup> C (‰)
UB-4379	24/06/1999	-23.79
UB-4380	24/06/1999	-23.79
UB-4381	24/06/1999	-24.83
UB-4382	24/06/1999	-24.68
UB-4383	24/06/1999	-24.58
UB-4384	24/06/1999	-24.61
UB-4385	17/06/1999	-23.98
UB-4386	17/06/1999	-24.12
UB-4387	17/06/1999	-23.81
UB-4388	26/05/1999	-23.85
UB-4389	30/04/1999	-24.31
UB-4390	30/04/1999	-24.22
UB-4391	30/04/1999	-24.34
UB-4392	30/04/1999	-24.34
UB-4393	21/04/1999	-24.72
UB-4394	21/04/1999	-24.71
UB-4395	21/04/1999	-24.98
UB-4396	21/04/1999	-25.09
UB-4397	21/04/1999	-25.01
UB-4398	21/04/1999	-25.42
UB-4399	29/02/1999	-25.98
UB-4400	19/02/1999	-25.96
UB-4401	19/02/1999	-25.56
UB-4402	19/02/1999	-25.50
UB-4403	19/02/1999	-25.73
UB-4404	19/02/1999	-25.81
UB-4405	19/02/1999	-25.73
UB-4406	11/02/1999	-25.90
UB-4407	11/02/1999	-25.23
UB-4408	11/02/1999	-25.21
UB-4409	/02/ 999	-25.46
UB-4410	11/02/1999	-25.46
UB-4616	07/03/2001	-24.45
UB-4617	22/03/2001	-24.43
UB-4618	22/03/2001	-24.60
UB-4619	22/03/2001	-24.85
UB-4620	22/03/2001	-23.95
UB-4621	22/03/2001	-24.25
UB-4622	22/03/2001	-24.54
UB-4623	22/03/2001	-24.54
UB-4624	22/03/2001	-24.77
UB-4625	22/03/2001	-24.65
UB-4626	22/03/2001	-24.54

Table 15:  $\delta^{3}C$  values on wood cellulose prepared and measured in Belfast before October 2001

UB-4627	02/04/2001	-24.98
UB-4628	02/04/2001	-25.18
UB-4629	02/04/2001	-24.99
UB-4630	02/04/2001	-25.06
Laboratory code	Date measured	δ <sup>13</sup> C (‰)
UB-4631	02/04/2001	-24.98

 $Mean = 24.82 \pm 0.03$ 

Standard deviation (=square root of sample variance) = 0.61

# Table 16: $\delta^{I3}C$ values on wood cellulose prepared and measured in Belfast after October 2001

Laboratory code	Date measured	δ <sup>13</sup> C (‰)
UB-4933	22/07/2003	-26.23
UB-4934	22/07/2003	-26.23
UB-4935	22/07/2003	-25.84
UB-4936	22/07/2003	-25.84
UB-4937	06/08/2003	-24.87
UB-4938	06/08/2003	-24.87
UB-4939	06/08/2003	-23.87
UB-4940	06/08/2003	-23.87
UB-4941	06/08/2003	-24.81
UB-4942	06/08/2003	-24.81
UB-4943	06/08/2003	-24.38
UB-4944	06/08/2003	-24.38
UB-4945	05/09/2003	-25.28
UB-4946	05/09/2003	-25.28
UB-4947	05/09/2003	-24.68
UB-4948	05/09/2003	-24.68
UB-4949	18/09/2003	-25.39
UB-4950	18/09/2003	-25.39
UB-4951	18/09/2003	-24.88
UB-4952	18/09/2003	-24.88
UB-6107	07/04/2004	-25.15
UB-6108	07/04/2004	-25.15
UB-6109	19/03/2004	-25.00
UB-6110	06/07/2005	-25.78
UB-6111	06/07/2005	-26.33
UB-6112	18/09/2005	-25.93
UB-6113	06/07/2005	-26.94
UB-6114	18/09/2005	-25.86
UB-6115	04/10/2005	-25.00
UB-6116	04/10/2005	-25.00
UB-6117	18/09/2005	-26.19
UB-6118	18/09/2005	-26.30
UB-6119	18/09/2005	-25.77

UB-6120	18/09/2005	-25.80
UB-6121	18/09/2005	-25.66
UB-6122	18/09/2005	-25.71

Mean =  $-25.34 \pm 0.03$ 

Standard deviation (=square root of sample variance) = 0.71

The reproducibility of  $\delta^{13}$ C values measured in New Zealand on replicate samples of bone gelatinized at the Rafter Radiocarbon Laboratory is demonstrated by a series of replicate measurements (Table 17). All pairs are statistically consistent (at 95% confidence).

Table 17:  $\delta^{13}C$  values measured at Isotrace New Zealand from replicate samples of bone separately gelatinized at the Rafter Radiocarbon Laboratory (T'(5%)=3.8; v=1 for all pairs)

Laboratory Number	Grave	$\delta^{_{13}}$ C (‰) Total	$\delta^{13}$ C (‰) Total	Τ'
		error	error	
UB-4965/UB-6344	Appledown Compton, grave 117	-20.60±0.32	-20.2±0.36	0.7
UB-4879	Lundenwic: ROH90, grave 31	-20.80±0.32	-20.18±0.32	1.9
UB-4930/UB-6346	St Peter's Tip, grave 42	-19.40±0.32	-19.00±0.36	0.7
UB-4925	St Peter's Tip, grave 68	-19.47±0.32	-19.28±0.32	0.2
UB-4924/UB-6534	St Peter's Tip, grave 113	-19.70±0.32	-19.40±0.36	0.4
UB-4927	St Peter's Tip, grave 263	-19.76±0.32	-19.67±0.32	0.0
UB-6472	Dover Buckland, grave 222	-19.80±0.36	-20.08±0.32	0.3
UB-6474	Dover Buckland, grave 264	-19.70±0.36	-19.97±0.32	0.3
UB-4923	Edix Hill, grave 7	-20.30±0.32	-20.35±0.32	0.0
UB-4511	Edix Hill, grave 90	-20.10±0.32	-20.65±0.32	1.5
UB-4886/UB-6345	Melbourne, SK 1204 SG77	-20.00±0.32	-19.50±0.36	1.1
UB-4885	Melbourne, InL 1189 SG78	-20.10±0.32	-20.49±0.32	0.7
UB-4884	Melbourne, SK1188 SG79	-20.40±0.32	-20.38±0.32	0.0
UB-4984	Lechlade, grave 18	-20.40±0.32	-20.60±0.32	0.2
UB-4683	Lechlade, grave 40	-19.70±0.36	-20.47±0.32	2.6

It is apparent, nonetheless, that the  $\delta^{13}$ C measurements on bone samples from October 2001 gradually show a greater offset between Belfast and New Zealand (Fig 24). In 2005 the VG 602e Micromass in the Belfast Radiocarbon Laboratory was serviced and found to be poorly focused. The mass spectrometer focus problem combined with sulphur contamination in some of the bone samples caused measurements on such samples to be inaccurate. Unfortunately material was not available to allow a sufficient number of remeasurements to be made to demonstrate this unequivocally.

A bias in measured  $\delta^{13}$ C values of the magnitude shown in Figure 24 would make radiocarbon ages calculated using the anomalous values substantively too young. For this reason, the radiocarbon ages reported here have been calculated using  $\delta^{13}$ C values measured in Belfast only for samples converted to benzene before October 2001.

Radiocarbon ages for samples processed after October 2001 cannot be calculated on the basis of these values. We have seen that the  $\delta^{13}$ C values on the dated protein measured in Belfast and New Zealand are in very good agreement before October 2001 (mean difference = 0.07±0.54‰). Unfortunately  $\delta^{13}$ C values on this material measured in New Zealand are only available for 79% of the samples run after October 2001, and so these

cannot be used to calculate radiocarbon ages for all the affected samples. For this reason radiocarbon ages for these samples have been calculated using the  $\delta^{13}$ C values from bone gelatin prepared at the Rafter Radiocarbon Laboratory and measured at Isotrace New Zealand . These values have been offset by the mean difference between the  $\delta^{13}$ C values measured in Belfast on the dated protein and the  $\delta^{13}$ C values measured in New Zealand on bone gelatin prepared at Rafter from the same samples in the period before October 2001. These datasets are also in good agreement (Fig 25) and, although the mean difference between them is slightly larger ( $-0.25\pm0.39\%$ ; Table 18), the use of  $\delta^{13}$ C values from bone gelatinized in New Zealand rather than from the dated protein in practice shifts the calculated radiocarbon ages by less than a year. The total measurement error on the  $\delta^{13}$ C values used to calculate these radiocarbon ages was taken as the square root of the sum of the squares of the total error estimate quoted by New Zealand and the standard deviation on the mean offset.

The  $\delta^{13}$ C values used to calculate all the radiocarbon ages reported here are shown in Table 11. Stable isotope values more appropriate for dietary studies are reported in Beavan *et al* (2011).

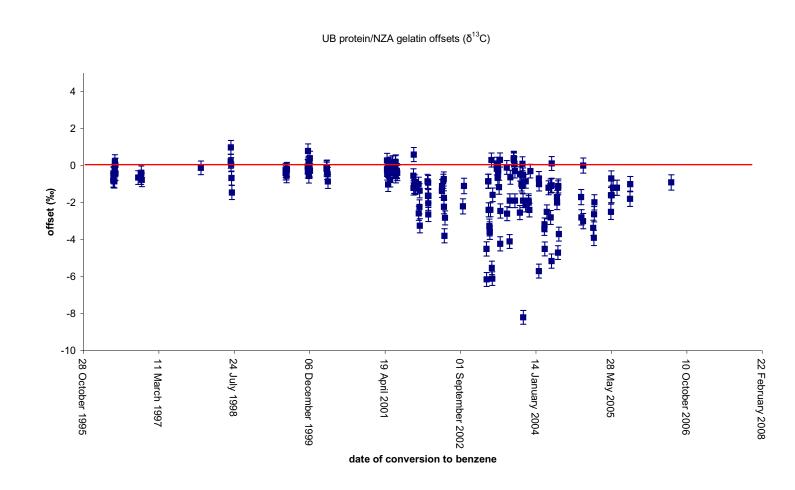


Figure 25:  $\delta^{I3}C$  values measured at Belfast on the protein extracted for radiocarbon dating and  $\delta^{I3}C$  values measured by Isotrace New Zealand on gelatin prepared by the Rafter Radiocarbon Laboratory from the same samples

		Belfast	Belfast		nd
Laboratory		δ <sup>13</sup> C(‰)	error	δ <sup>13</sup> C(‰)	total error
Number			(‰)		(‰)
UB-4039	Buttermarket, grave 3871	-20.9	0.2	-20.I	0.31
UB-4040	Buttermarket, grave 3897	-20.1	0.2	-19.7	0.31
UB-4041	Buttermarket, grave 249	-20.3	0.2	-19.8	0.31
UB-4042	Buttermarket, grave 1674	-21.3	0.2	-20.5	0.31
UB-4043	Buttermarket, grave 2365	-20.9	0.2	-20.I	0.31
UB-4044	Buttermarket, grave 4152	-20.3	0.2	-19.8	0.31
UB-4045	Buttermarket, grave 4307	-19.5	0.2	-19.2	0.31
UB-4046	Buttermarket, grave 4344	-20.2	0.2	-20.1	0.31
UB-4047	Buttermarket, grave 4431	-19.7	0.2	-19.9	0.31
UB-4048	Buttermarket, grave 4926	-20.1	0.2	-20.1	0.31
UB-4049	Buttermarket, grave 4979	-20.5	0.2	-20.I	0.31
UB-4074	Buttermarket, grave 2297	-20.6	0.2	-20.0	0.31
UB-4075	Buttermarket, grave 3898	-20.0	0.2	-19.4	0.31
UB-4076	Buttermarket, grave 4269	-20.4	0.2	-20.0	0.31
UB-4077	Buttermarket, grave 4275	-21.0	0.2	-20.2	0.31
UB-4210	Buttermarket, grave 1760	-19.9	0.2	-19.8	0.31
UB-4306	Known-age bone, sample 1	-18.6	0.2	-18.8	0.32
UB-4307	Known-age bone, sample 2	-18.6	0.2	-19.6	0.32
UB-4308	Known-age bone, sample 3	-18.3	0.2	-18.4	0.32
UB-4309	Known-age bone, sample 4	-18.3	0.2	-17.6	0.32
UB-4310	Known-age bone, sample 5	-20.3	0.2	-18.8	0.32
UB-4440	Barton on Humber BH14	-20.0	0.2	-19.8	0.32
UB-4441	Barton on Humber BH15	-20.1	0.2	-19.7	0.32
UB-4442	Barton on Humber BH16	-20.4	0.2	-20.0	0.32
UB-4443	Barton on Humber BH17	-19.6	0.2	-19.1	0.32
UB-4444	Barton on Humber BH 18	-19.9	0.2	-19.7	0.32
UB-4445	Barton on Humber BH 19	-19.9	0.2	-19.7	0.32
UB-4501	Lechlade: grave 14	-20.0	0.2	-19.9	0.32
UB-4502	Lechlade: grave 138	-20.4	0.2	-20.1	0.32
UB-4503	Lechlade: grave 148	-19.3	0.2	-20.1	0.32
UB-4504	Lechlade: grave 179	-20.3	0.2	-20.2	0.32
UB-4505	Lechlade: grave 172/1	-20.2	0.2	-20.3	0.32
UB-4506	Lechlade: grave 172/2	-20.2	0.2	-20.0	0.32
UB-4507	Lechlade: grave 187	-20.2	0.2	-19.6	0.32
UB-4508	Edix Hill: grave 12	-20.2	0.2	-20.0	0.32
UB-4509	Edix Hill: grave 33	-20.1	0.2	-20.2	0.32
UB-4510	Edix Hill: grave 48	-20.2	0.2	-20.6	0.32
UB-4511	Edix Hill: grave 90	-20.3	0.2	-20.1	0.32
UB-4512	Edix Hill: grave 91	-20.5	0.2	-20.2	0.32
UB-4549	Marina Drive: grave C7	-20.6	0.2	-20.4	0.32

Table 18:  $\delta^{I3}C$  values measured in Belfast on the bone protein extracted for radiocarbon dating and  $\delta^{I3}C$  values measured at Isotrace New Zealand on bone gelatin prepared from the same samples at the Rafter Radiocarbon Laboratory

UB-4550	Marina Drive: grave El	-20.0	0.2	-19.9	0.32
UB-4551	Marina Drive: grave E2	-20.3	0.2	-20.2	0.32
UB-4552	Marina Drive: grave E3	-20.5	0.2	-20.1	0.32
UB-4553	Marina Drive: grave D10	-20.8	0.2	-20.3	0.32
		Belfast		New Zealand	
Laboratory		$\delta^{13}C(\infty)$	error	δ <sup>13</sup> C(‰)	total error
Number			(‰)		(‰)
UB-4554	Marina Drive: grave F2	-20.8	0.2	-19.9	0.32
UB-4641	West Heslerton, grave 71	-20.0	0.2	-19.8	0.32
UB-4642	West Heslerton, grave 72	-19.9	0.2	-20.2	0.32
UB-4643	Castledyke South, grave 5	-20.3	0.2	-19.9	0.32
UB-4644	Castledyke South, grave 15	-21.3	0.2	-20.3	0.32
UB-4645	Castledyke South, grave 54	-19.9	0.2	-19.9	0.32
UB-4646	Castledyke South, grave 84	-20.2	0.2	-19.9	0.32
UB-4647	Barton on Humber BH23	-18.8	0.2	-18.8	0.32
UB-4648	Barton on Humber BH25	-19.9	0.2	-19.8	0.32
UB-4649	Barton on Humber BH27	-20.1	0.2	-20.0	0.32
UB-4650	Barton on Humber BH28	-19.9	0.2	-19.2	0.32
UB-4651	Barton on Humber BH29	-19.4	0.2	-19.1	0.32
UB-4652	Barton on Humber BH31	-19.4	0.2	-19.5	0.32
UB-4653	Barton on Humber BH33	-20.0	0.2	-19.9	0.32
UB-4654	Barton on Humber BH34	-19.7	0.2	-19.6	0.32
UB-4655	Barton on Humber BH35	-19.8	0.2	-19.9	0.32
UB-4656	Barton on Humber BH36	-20.0	0.2	-19.9	0.32
UB-4657	Barton on Humber BH37	-19.5	0.2	-19.4	0.32
UB-4658	Barton on Humber BH38	-19.5	0.2	-19.7	0.32
UB-4659	Barton on Humber BH39	-19.4	0.2	-19.3	0.32
UB-4660	Barton on Humber BH40	-19.5	0.2	-19.7	0.32
UB-4661	Barton on Humber BH41	-20.4	0.2	-19.8	0.32
UB-4662	Barton on Humber BH72	-20.0	0.2	-19.8	0.32
UB-4663	Barton on Humber BH73	-19.2	0.2	-18.9	0.32
UB-4682	Westgarth Gardens: grave 66	-20.2	0.2	-19.9	0.36
UB-4683	Lechlade: grave 40	-20.1	0.2	-19.7	0.36
UB-4705	West Heslerton: 002BA 00606	-20.9	0.2	-20.3	0.32
UB-4706	West Heslerton: 002BA00536	-21.1	0.2	-19.9	0.32
UB-4707	Edix Hill: grave 79	-19.7	0.2	-20.3	0.32
UB-4708	Edix Hill: grave 83	-21.3	0.2	-20.2	0.32
UB-4709	Edix Hill: grave 14	-21.3	0.2	-20.4	0.32
UB-4719	Barton on Humber BH89	-20.8	0.2	-19.6	0.32
UB-4720	Barton on Humber BH90	-20.5	0.2	-19.4	0.32
UB-4727	Gally Hills: primary burial	-21.3	0.2	-20.3	0.32
UB-4728	Mill Hill: grave 64	-22.1	0.2	-19.5	0.32
UB-4729	Mill Hill: grave 68	-21.7	0.2	-19.5	0.32
UB-4730	Mill Hill: grave 79	-20.5	0.2	-19.1	0.32
UB-4731	Mill Hill: grave 93	-23.1	0.2	-19.8	0.32
UB-4732	Mill Hill: grave 94	-20.6	0.2	-19.7	0.32
UB-4733	Mill Hill: grave 95	-20.8	0.2	-19.7	0.32

UB-4734	Mill Hill: grave 105C	-21.2	0.2	-20.3	0.32
UB-4735	Berinsfield: grave 22	-21.9	0.2	-19.9	0.32
UB-4736	Berinsfield: grave 28	-22.6	0.2	-20.0	0.32
UB-4739	Berinsfield: grave 134/1	-21.9	0.2	-20.3	0.32
UB-4796	Known-age bone, sample 6	-21.0	0.2	-19.6	0.32
UB-4797	Known-age bone, sample 7	-20.6	0.2	-19.5	0.32
UB-4798	Known-age bone, sample 8	-20.0	0.2	-19.9	0.32
UB-4799	Known-age bone, sample 9	-19.3	0.2	-19.9	0.32
OB-4777	Kilown-age bone, sample /	Belfast	0.2	New Zeala	
Laboratory		$\delta^{13}C(\%)$	error	$\delta^{13}C(\infty)$	total error
Number		0 (/00)	(‰)	0 C(/00)	(‰)
UB-4800	Known-age bone, sample 10	-21.2	0.2	-19.4	0.32
UB-4801	Known-age bone, sample 11	-21.8	0.2	-19.6	0.32
UB-4802	Known-age bone, sample 12	-22.8	0.2	-19.0	0.32
UB-4803	Known-age bone, sample 13	-22.6	0.2	-19.8	0.32
UB-4835	Appledown Compton, grave 134	-21.3	0.2	-20.2	0.32
UB-4836	Westgarth Gardens, grave 27	-21.3	0.2	-20.2	0.36
UB-4882	Melbourne: SKI 187 SG80	-22.0	0.2		0.30
UB-4883	Melbourne: SK1038, SG95		0.2	-20.1	0.32
UB-4884	Melbourne: SKI 188 SG79	-26.3	0.2	-20.1	0.32
		-21.2		-20.4	
UB-4885	Melbourne, InLI 189 SG78	-20.9	0.2	-20.1	0.32
UB-4886	Melbourne: SK1204 SG77	-22.4	0.2	-20.0	0.32
UB-4887	Melbourne: SK 1229 SG82	-22.7	0.2	-20.3	0.32
UB-4888	Melbourne: SK1271 SG89	-23.9	0.2	-20.3	0.32
UB-4889	Melbourne: SK1293, SG69	-23.6	0.2	-20.2	0.32
UB-4890	Melbourne: SK1307 SG75	-23.6	0.2	-20.3	0.32
UB-4896	Claydon Pike FCPA	-25.0	0.2	-19.5	0.31
UB-4897	Claydon Pike FCPB	-25.9	0.2	-19.8	0.31
UB-4898	Claydon Pike FCPC	-21.2	0.2	-19.6	0.31
UB-4907	Bloodmoor Hill: F165 (2304)	-20.0	0.2	-19.9	0.32
UB-4908	Bloodmoor Hill: F173 (1440)	-20.2	0.2	-20.3	0.32
UB-4909	Bloodmoor Hill: F185 (1520)	-20.6	0.2	-20.3	0.32
UB-4910	Bloodmoor Hill: F236 (2280)	-20.6	0.2	-20.2	0.32
UB-4911	Bloodmoor Hill: F241 (2238)	-20.8	0.2	-20.1	0.32
UB-4912	Bloodmoor Hill: F256 (2259)	-20.7	0.2	-20.5	0.32
UB-4913	Bloodmoor Hill: F257 (2260)	-20.4	0.2	-20.7	0.32
UB-4914	Bloodmoor Hill: F261 (2261)	-24.5	0.2	-20.3	0.32
UB-4915	Bloodmoor Hill: F364 (3417)	-22.6	0.2	-20.1	0.32
UB-4916	Bloodmoor Hill: F265 (2269)	-20.8	0.2	-19.6	0.32
UB-4920	replicate of UB-4727	-20.1	0.2	-20.2	0.32
UB-4921	Mill Hill: grave 81	-20.4	0.2	-20.3	0.32
UB-4922	replicate of UB-4510	-23.2	0.2	-20.6	0.32
UB-4923	Edix Hill: grave 7	-24.4	0.2	-20.3	0.32
UB-4924	St Peter's Tip: grave 113	-21.1	0.2	-19.7	0.32
UB-4925	St Peter's Tip: grave 68	-20.1	0.2	-19.47	0.32
UB-4927	St Peter's Tip: grave 263	-19.4	0.2	-19.47	0.32
UB-4928					
UD-7720	St Peter's Tip: grave 250	-19.7	0.2	-20.I	0.32

UB-4929	St Peter's Tip: grave 194	-19.7	0.2	-20	0.32
UB-4930	St Peter's Tip: grave 42	-19.7	0.2	-20	0.32
UB-4931	St Peter's Tip: grave 318		0.2		0.32
UB-4958	Dover Buckland: grave 375	-21.9	0.2	-20	0.32
UB-4959	Dover Buckland: grave 373	-22.3 -20.7	0.2		0.32
UB-4939 UB-4960	Dover Buckland: grave 391A Dover Buckland: grave 391B		0.2	-20.3	0.32
UB-4960 UB-4961	_	-21.0	0.2	-20	0.32
UB-4961 UB-4962	St Peter's Tip: grave 8	-20.6		-19.5	
	St Peter's Tip: grave 196	-19.8	0.2	-19.9	0.32
UB-4963	St Peter's Tip: grave 208	-21.7	0.2	-19.8	0.32
UB-4964	Coddenham: grave 308	-20.9		-20.4	0.32
UB-4965	Appledown Compton: grave 117	-28.8	0.2	-20.6	0.32
UB-4975	Aston Clinton: grave 12	-22.9	0.2	-20.77	0.32
		Belfast		New Zealar	
Laboratory		δ <sup>13</sup> C(‰)	error	δ <sup>13</sup> C(‰)	total error
Number	Ford, Laverstock: barrow 2	21.2	(‰) 0.2	20.25	(‰) 0.32
UB-4976		-21.2		-20.35	
UB-4981	Lechlade: grave 183	-22.2	0.2	-20.3	0.32
UB-4982	Lechlade: grave 155	-22.7	0.2	-20.7	0.32
UB-4984	Lechlade: grave 18	-22.8	0.2	-20.4	0.32
UB-4985	Westgarth Gardens: grave 11	-21.1	0.2	-20.8	0.32
UB-5208	Appledown Compton, grave 107	-22.1	0.2	-20.3	0.36
UB-6031	Sutton Courtenay, DRST02 163	-24.5	0.2	-18.8	0.32
UB-6032	St Peter's Tip, grave 73A	-21.6	0.2	-20.9	0.32
UB-6033	West Heslerton, grave 113	-21.8	0.2	-20.8	0.32
UB-6034	Castledyke South, grave 120	-24.3	0.2	-20.9	0.32
UB-6035	Castledyke South, grave 96	-24.5	0.2	-21.3	0.32
UB-6036	Castledyke South, grave 13	-20.6	0.2	-20.9	0.32
UB-6037	Castledyke South, grave 134	-25.1	0.2	-20.6	0.32
UB-6038	Castledyke South, grave 183	-23.5	0.2	-21	0.32
UB-6039	Castledyke South, grave 94	-21.7	0.2	-20.5	0.32
UB-6040	Castledyke South, grave 53	-22.2	0.2	-21.1	0.32
UB-6041	Castledyke South, grave 182	-23.3	0.2	-20.5	0.32
UB-6042	Castledyke South, grave 88	-21.4	0.2	-20.3	0.32
UB-6142	Known age bone - sample 14	-22.8	0.2	-17.6	0.32
UB-6143	Known age bone - sample 15	-18.3	0.2	-18.4	0.32
UB-6144	Known age bone - sample 16	-19.7	0.2	-18.6	0.32
UB-6179	Riccall Landing, 1974.121 SKE	-22.1	0.2	-20.4	0.32
UB-6180	Riccall Landing, 1974.121 SK18	-22.4	0.2	-20.1	0.32
UB-6181	Riccall Landing, 1985.11 SK1	-22.4	0.2	-20.4	0.32
UB-6182	Riccall Landing, 1985.11 SK17		0.2		0.32
UB-6183	Riccall Landing, 1976.27 SKA	-21.1	0.2	-19.9	0.32
	0	-21.5		-20.4	
UB-6184	Riccall Landing, 1974.121 SK4	-23.7	0.2	-20	0.32
UB-6345	Melbourne: SK1204 SG77	-22.3	0.2	-19.5	0.36
UB-6346	St Peter's Tip: grave 42	-20.7	0.2	-19	0.36
UB-6347	Lakenheath, ERL 104 4222	-22.8	0.2	-19.8	0.36
UB-6348	Lakenheath, ERL 104 4206	-22.6	0.2	-22.6	0.36
UB-6394	Crickley Hill CH71 F370	-25.3	0.2	-21.9	0.36

UB-6395	Crickley Hill 3396	-26.0	0.2	-22.1	0.36
UB-6396	Crickley Hill CH77 4517	-24.7	0.2	-22.1	0.36
UB-6397	Crickley Hill CH77 4582	-24.3	0.2	-22.3	0.36
UB-6472	Dover Buckland, Grave 222	-22.3	0.2	-19.8	0.36
UB-6473	Dover Buckland, Grave 250	-21.4	0.2	-19.8	0.36
UB-6474	Dover Buckland, Grave 264	-20.9	0.2	-19.7	0.36
UB-6475	Dover Buckland, Grave 323	-20.5	0.2	-19.8	0.36
UB-6476	Dover Buckland, Grave 339	-21.3	0.2	-19.7	0.36
UB-6477	Dover Buckland, Grave 414	-21.0	0.2	-19.8	0.36
UB-6478	St Peter's Tip, Grave 360	-21.1	0.2	-20.1	0.36
UB-6479	Mill Hill, Grave 40	-20.7	0.2	-19.5	0.36
UB-6534	St Peter's Tip: grave 113	-20.0	0.2	-19.4	0.36

### pre-October 2001

Number of comparisons = 70 Average difference = -0.25Average standard deviation in difference = 0.39Mean difference =  $-0.25 \pm 0.05$ Standard deviation (=square root of sample variance) = 0.39k = Standard deviation/Average standard deviation in difference = 1.03

post-October 2001 Number of comparisons = 115 Average difference = -1.65Average standard deviation in difference = 2.59 Mean difference =  $-1.63 \pm 0.04$ Standard deviation (=square root of sample variance) = 2.7 k = Standard deviation/Average standard deviation in difference = 7.04

## II CONCLUSIONS

This report describes the laboratory techniques used for dating the radiocarbon samples processed at the Belfast Radiocarbon Laboratory for the Anglo-Saxon chronology project. Procedures for fractionation correction and age calculation are also discussed. As part of this project a bespoke section of calibration curve covering the early Anglo-Saxon period was produced. Details of the radiocarbon measurements and the dated tree-ring series from which the samples were taken are also described.

The samples were measured over an eight-year period (1999–2006). The reproducibility of the radiocarbon measurements over this extended period is demonstrated by the excellent agreement between the decadal replicate pairs of known-age wood (Fig 12), and by the good agreement between a limited number of replicate bone samples (Section 8).

The accuracy of these radiocarbon measurements is demonstrated by the similarity of the new calibration data with those produced previously by Pearson *et al* (1986) (Fig 14) and Stuiver and Becker (1986, corrected as reported in Stuiver *et al* (1998a)) (Fig 15). More generally, measurement accuracy is demonstrated by the results reported for international inter-comparison exercises undertaken during this time (Table 10).

The tree-ring measurements were made quasi-simultaneously with the bone samples dated for this project, and so will provide the most accurate calibration of these samples (as, for example, there are no laboratory offsets to be considered). However, these

results will be incorporated for general use in the next international radiocarbon calibration curve (IntCal).

Archaeological evidence also provides some independent confirmation of the relative accuracy of the calibrated dates. Six dated samples can be related to others stratigraphically (Fig 23). In all cases the results are in good agreement with the excavated sequence.

It is more difficult to assess whether the dated bone protein in all cases derived from a purely terrestrial reservoir, as required if measurements on human bone are to be converted accurately to the calendar timescale by tree-ring calibration. A replicate pair of measurements on a horse and human buried together at Lakenheath, however, are statistically consistent (UB-6347–8). This issue is discussed further in Beavan *et al* (2011).

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### NOTES

- <sup>1</sup> A third measurement was made on excess pre-treated cellulose from this sample in July 2006. This result is 1283±20 BP (UB-5232); δ<sup>13</sup>C -25.2±0.2‰.
- <sup>2</sup> The result for the bi-decade centred on AD 710 produced by Pearson *et al* (1986) is 1282±17 BP. This has been confirmed by reference to the primary laboratory archive as the error was previously omitted from the published dataset due to a printing error.
- <sup>3</sup> The  $\delta^{13}$ C values reported in this table are those used in the calculation of the radiocarbon age (see Section 9). Stable isotope values appropriate for dietary studies are reported and discussed in Beavan *et al* (2011)
- <sup>4</sup> Replicate measurements are also available from two burial mounds at Sutton Hoo, Suffolk (Carver 2005, 54–5). The two measurements on bone collagen from a human inhumation and a horse burial from beneath mound 17 are not statistically consistent (UB-4422 and UB-4423; T'=6.5; T'(5%)=3.8; v=1), almost certainly because of the incomplete removal of a chemical consolidant from the sample of human bone. Results from a sample of beeswax from the iron lamp in the ship burial beneath mound 1 (BM-640) and from a fragment of oak interpreted as part of the burial chamber (BM-688) are also statistically inconsistent (T'=7.1; T'(5%)=3.8; v=1). Burleigh *et al* (1976, 22) consider BM-688 to be anomalously young because of contamination by younger humic material.



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